AMERICAN SOCIETY
for
TESTING MATERIALS

Reg. U. S. Pat. Off.

PROCEEDINGS
of the
FORTY-FOURTH ANNUAL MEETING

Held at Chicago, Ill.

June 23–27, 1941

VOLUME 41

COMMITTEE REPORTS

TECHNICAL PAPERS

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SUMMARY OF PROCEEDINGS OF THE FORTY-FOURTH ANNUAL MEETING

CHICAGO, ILL., JUNE 23–27, 1941

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards and Tentative Standards appearing in the annual reports of the standing committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee. The new and revised standards and tentative standards are published in the 1941 Supplement to the Book of A.S.T.M. Standards, Parts I, II, and III. Except as noted, the reports and papers appear in this volume.

This summary of the Forty-fourth Annual Meeting of the American Society for Testing Materials, held at the Palmer House, Chicago, Ill., June 23–27, 1941, is a record of the transactions of the meeting including the actions taken on the various recommendations submitted by the standing committees. In all, 17 sessions were held, as well as the Sixth Exhibit of Testing Apparatus and Related Equipment, the Fourth A.S.T.M. Photographic Exhibition and Competition, and the Twenty-fourth Annual A.S.T.M. Golf Tournament. The exhibit of testing apparatus and the photographic exhibition are described in some detail in the August ASTM BULLETIN.

The *Proceedings* are set forth session by session. There were 62 reports and 68 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentative standards and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentative standards as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard," it is understood that this indicates approval of the annual meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" are self evident as indicating acceptance by the Society at the annual meeting for publication as tentative.

While all of the items on the program are recorded under the particular session in which they were presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of the reports together with the page reference where the action thereon is recorded.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on September 2, 1941, and all actions were approved.

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The registered attendance of the meeting is as follows: Members present or represented, 969; committee members, 193; guests, 391; total, 1553; ladies, 100.

FORMAL OPENING SESSION
PRESIDENTIAL ADDRESS
TUESDAY, JUNE 24, 10.30 A.M.

Session Co-Chairmen: President W. M. Barr; Past-President A. E. White.

President Barr, in opening the Forty-fourth Annual Meeting, referred briefly to the outstanding features of this meeting which were of particular significance, mentioning specifically the Sixth Exhibit of Testing Apparatus and Related Equipment, and the Fourth Photographic Exhibition, the session on Subways and Superhighways to be held jointly with the Western Society of Engineers, and the plans made for the meeting by the Chicago Committee, including the informal reception and officers' night scheduled for Wednesday evening. He also referred briefly to the many activities which the Society had fostered through its standing committees during the past year, as evidenced by the large number of reports to be presented at the technical sessions. The President stated that from the advance attendance records, the number of committee meetings scheduled, and other activities this meeting promised to be the most successful that the Society had yet held.

President Barr then presented the honorary chairman, H. H. Morgan, and the chairman, E. R. Young, of the Chicago Committee on Arrangements. On behalf of the Chicago Committee the honorary chairman, Mr. Morgan, extended a cordial welcome to the Society membership, and expressed the wish that the plans made by the local committee would contribute to a most successful annual meeting.

The chairman of the Chicago Committee, Mr. Young, then outlined briefly the plans and arrangements that had been made for the entertainment of the members and guests, particularly for the ladies, during the meeting. He said that no effort had been spared by the committee to make this a grand occasion. He called particular attention to three features planned during the meeting, namely, the joint session with the Western Society of Engineers devoted to subways and superhighways, the informal reception on Wednesday afternoon following the Marburg Lecture, at which the members would have an opportunity to meet the retiring and the new officers of the Society, to be concluded by the officers' night at the Exhibit of Testing Apparatus.

Committee E-9 on Research:

Report presented from manuscript by G. F. Jenks, chairman.

Committee E-10 on Standards:

Report presented by R. P. Anderson, chairman.

The chairman then introduced Mr. J. H. Van Deventer, President and Editor of *The Iron Age*, who presented a talk on "Mobilizing Materials for Defense."[2]

Past-President A. E. White then assumed the chair.

Executive Committee:

This report, presented by the Secretary-Treasurer, C. L. Warwick, reviewed at some length the present and potential relationship of the A.S.T.M. to the various phases of the national defense program. In this connection Mr. Warwick discussed briefly the considerations being given by the Executive Committee of the Society to a procedure intended to expedite the approval and publication of revisions of the Society's standards made necessary by the present emergency, and which at the same time would provide adequate safeguards in their promulgation. The report also covered general Society activities, including membership, publications, finances, and administrative matters relating to committee activities, cooperative activities with other engineering societies, and engineering relations between the American Republics.

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[2] Published in ASTM BULLETIN, No. 111, August, 1941, p. 19.

The recommendation in the report for a revision in Article III, Section 2, of the By-laws with reference to the method of selecting members of the Nominating Committee, providing for their appointment at the October quarterly meeting of the Executive Committee instead of the January meeting, was approved for reference to letter ballot of the Society.[3]

Recognition of Forty-Year Member:

The chairman announced that in accordance with the action taken by the Executive Committee, a certificate for having attained 40 years of membership in the Society was being awarded this year to Mr. C. N. Forrest, of the Barber Asphalt Corp. Presentation of the certificate to Mr. Forrest was then made by President Barr.

Election of Officers:

The chairman then recognized J. de N. Macomb, chairman of the Committee of Tellers, who reported the results of the letter ballot on election of officers. Of the 1049 votes cast, the results were as follows:

For President, to serve for one year:
  G. E. F. Lundell, 1049 votes.
For Vice-President, to serve for two years:
  Dean Harvey, 1049 votes.
For Members of the Executive Committee, to serve for two years:
  Arthur W. Carpenter, 1037 votes.
  T. A. Fitch, 1027 votes.
  C. D. Hocker, 1029 votes.
  J. L. Miner, 1029 votes.
  E. W. Upham, 1036 votes.

The chairman then introduced the newly elected members of the Executive Committee, requesting that they rise as their names were called.

At the request of the chairman, Vice-President-Elect, Dean Harvey, was escorted to the chair. He expressed sincere appreciation of the honor of his election and said that he would make every effort to justify the confidence placed in him by the membership.

The chairman then requested Past-Presidents T. G. Delbridge and H. F. Moore to escort to the chair the President-Elect, G. E. F. Lundell, who made the following remarks:

> I wish to express my deep appreciation for the honor which the Society has conferred on me. In the difficult year to come, I shall do my best, and so, I am sure, will the membership, for I know of no other technical society with which I have contact, in which the average member works so hard or has such personal contact with the affairs of the Society as he has with the affairs of the American Society for Testing Materials. As a result, the A.S.T.M. is particularly ready to meet the special demands which will undoubtedly be made on the members; nor do I know of any other society which possesses such a vast store of technical knowledge on which it can draw in emergency, and so it is with confidence that I ask your cooperation in the year ahead.

Presidential Address:

The chairman then presented the President, W. M. Barr, who delivered the Presidential Address on "Speed, Specifications, and Safety." In his address, President Barr reviewed the tremendous improvement made in railroad transportation from the early days of the steam locomotive to the present fast high-powered streamlined Diesel and electric locomotives. These many improvements in speed and riding comfort introduced a number of problems requiring the development of new and higher grade materials to meet the increased stresses imposed by both weight and speed. He also pointed out that such advances have only been possible as the result of intensive research which required the constant revision of specifications and the establishment of many new standards. In stressing the many improvements in speed, specifications, and safety in the field of rail transportation, President Barr referred also to the fact that the same high standards of engineering research have perfected the automobile and made possible the tremendous strides in speed and safety of the airplane. In conclusion he prophesied that in the existing national emergency and in the years ahead, the safety of this nation and the future success of our entire economic life will be determined by the work of the research scientist and the production engineer in which the standardization and research work of the American Society for Testing Materials will have a prominent place.

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[3] This recommendation was approved by letter ballot of the Society which was canvassed on September 2, 1941.

SECOND SESSION—SUBWAYS AND SUPERHIGHWAYS

JOINT SESSION WITH WESTERN SOCIETY OF ENGINEERS

TUESDAY, JUNE 24, 2.30 P.M.

Session Co-Chairmen: F. H. Lane, President, W. S. E.; E. R. Young, Chairman, Chicago Committee.

This session, sponsored jointly by the Western Society of Engineers and the American Society for Testing Materials, featured two papers as follows:

The Measurement of Earth Pressures on the Chicago Subway[4]—Ralph B. Peck, Assistant Subway Engineer, Department of Subways and Superhighways, City of Chicago.

The General Plan of Superhighways—Charles DeLeuw, Acting Chief Engineer, Department of Subways and Superhighways, City of Chicago.

At the conclusion of the meeting a motion picture depicting construction details in the building of the Chicago Subway was shown.

THIRD SESSION—PLASTICS

TUESDAY, JUNE 24, 8 P.M.

Session Chairman: L. M. Currie.

Committee D-20 on Plastics:

Report presented, in the absence of the chairman, by W. A. Evans, secretary, and the following actions taken:

Accepted as Tentative:

Test for Deformation of Plastics and Electrical Insulating Materials Under Load at Elevated Temperatures, with a correction in the title by omission of the words "and Electrical Insulating Materials"

Preconditioning Plastics and Electrical Insulating Materials for Testing, recommended jointly with Committee D-9 on Electrical Insulating Materials

Test for Color Fastness of Plastics to Light

Revision of Test for Flammability of Plastics (D 568 - 40 T)

Revision of Test for Measuring Flow Temperatures of Thermoplastic Molding Materials (D 569 - 40 T)

Papers:

The following papers were presented:

Resistance of Plastics to Chemical Reagents—G. M. Kline, R. C. Rinker, and H. F. Meindl, presented from manuscript by Mr. Kline.

Mechanical Tests of Cellulose Acetate—William N. Findley, presented by the author.

Factors Influencing the Creep and Cold Flow of Plastics[5]—John Delmonte and W. Dewar, presented from manuscript by the author.

Method for Testing Odor-Taste Contamination Tendencies of Phenolic Plastic Closures[6]—A. Herman, presented by the author.

Shear Strength of Molded Plastic Materials—John Delmonte, presented by the author.

FOURTH SESSION—NON-FERROUS METALS

TUESDAY, JUNE 24, 8 P.M.

Session Chairman: Dean Harvey.

Committee B-2 on Non-Ferrous Metals and Alloys:

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[4] Published in ASTM BULLETIN, No. 111, August, 1941, p. 25.

[5] Published in ASTM BULLETIN, No. 112, October, 1941, p. 35.

[6] Published in ASTM BULLETIN, No. 111, August, 1941, p. 33.

Report presented, in the absence of the chairman, by G. H. Clamer, vice-chairman.

Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys:

Report presented by Dean Harvey, chairman, and the following actions taken:

Accepted as Tentative:

Test for Temper of Strip and Sheet Metals (Spring-Back Method)

Testing Lateral Wire for Grids of Electronic Devices

Testing Wire for Supports Used in Electronic Devices and Lamps

Adopted as Standard:

Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (B 113 – 38 T), as revised

Revision of Spec. for Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Electrical Heating Elements (B 82 – 39)

Revision of Spec. for Drawn or Rolled Alloy, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron, for Electrical Heating Elements (B 83 – 39)

Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors:

Report presented by J. H. Foote, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Bare Rope-Lay-Stranded and Bunch-Stranded Soft Copper Cables for Electrical Conductors, with additional changes, briefly summarized below, accepted by unanimous consent, subject to favorable letter ballot of Committee B-1:[7]

Section 1 on Scope is changed in wording to include both uncoated and coated copper wire, provided the coated wires have the same general weight and diameter as the coated wire. A provision has been added which allows the use of hard drawn wire in making up the cable which has been annealed after cabling, so that it is unnecessary to use soft annealed wire in very small sizes in cables involving a large number of very small conductors. The question of joints has been clarified and provision has been made for various types of joints for the various parts of the cable which seem to meet all requirements that have been presented. In the section covering the direction and length of lay of the cable, an additional paragraph has been added and a few modifications made in order to take care of all of the differences of opinion. Any absolute values of diameter or weight of completed cable have been eliminated. In addition, numerous editorial changes have been incorporated.

Adopted as Standard:

Revision of Spec. for Bare, Stranded Copper Cable, Hard, Medium-Hard, or Soft (B 8 – 39), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-1:[7]

Title.—Omit the word "Bare" from the title.

Section 1 (*a*).—Change to read as follows:

These specifications cover concentric-lay stranded cables made from round copper wires, either uncoated or coated with tin, for general use as electrical conductors.

Section 6 (*d*).—Change to read as follows:

(*d*) At the option of the purchaser, tension tests on hard-drawn and medium-hard-drawn wires before stranding may be waived, and completed hard-drawn and medium-hard-drawn cables may be tested as a unit. The tensile strength of bare cables so tested shall be at least 90 per cent of the total of the required minimum strengths of the component wires and the maximum tensile strength of cable made from medium-hard-drawn wires shall be not greater than the sum of the specified maximum tensile strengths of the component wires. The free length between grips of the test specimen shall be not less than 24 in., and care shall be taken to insure that the wires in the cable are evenly gripped during the test (Note 4).

Section 8.—In the last sentence change the phrase "shall be calculated and not assumed (Note 9)" to read "may be calculated if desired."

Note 9.—In the table referred to in this note a number of editorial changes were recommended by the committee in order to include more complete and later information.

Table II.—In the column headed "Area of Cross-Section, cir. mils" omit the asterisk

reference from 1,300,000; insert an asterisk before 1,250,000.

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[7] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee B-1, see pp. 163 and 164.

In the column headed "Diameter of Wires" under class D, change the wire size of the 1,700,000 cir. mils cable from "88.6 mils" to read "88.5 mils."

In the column headed "Diameter of Wires" under class B, change the wire size for the 3,500,000 cir. mils cable from "143.8 mils" to read "143.9 mils."

Revisions of Spec. for Soft or Annealed Copper Wire (B 3 - 39), and for Hot-Rolled Copper Rods for Electrical Purposes (B 49 - 39), subject to similar revisions being incorporated on the recommendation of Committee B-2 on Non-Ferrous Metals and Alloys in the Standard Specifications for Lake Copper Wire Bars (B 4 - 27), and for Electrolytic Copper Wire Bars (B 5 - 27).

Papers:

The following papers were presented:

The Properties of Certain Lead-Bearing Alloys—Albert J. Phillips, Albert A. Smith, Jr., and Paul A. Beck, presented by Mr. Smith.

Comparative Value of Arsenic, Antimony, and Phosphorus in Preventing Dezincification—W. Lynes, presented by the author.

The Tensile Properties of Some Copper Alloys—Cyril Stanley Smith and R. W. Van Wagner, presented by Mr. Smith.

Fatigue Tests on Some Copper Alloys—A. R. Anderson and Cyril Stanley Smith, presented by Mr. Smith.

FIFTH SESSION—NON-FERROUS METALS

(CONTINUED FROM FOURTH SESSION)

WEDNESDAY, JUNE 25, 9.30 A.M.

Session Co-Chairmen: T. S. Fuller; J. L. McCloud.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by J. R. Townsend, chairman, and the following actions taken:

Accepted as Tentative:

Revision of Spec. for Zinc-Base Alloy Die Castings (B 86 - 38 T)

It was announced that the committee at its meeting in Chicago had taken action to recommend the adoption as standard of the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 - 39 T) and to continue as tentative the Specifications for Lead- and Tin-Base Alloy Die Castings (B 102 - 39 T) subject to favorable letter ballot in the committee, this action having been taken since the two tentative standards have been standing in their present status for two years.

The following papers, appended to the report, were presented as indicated:

Finishes for Aluminum Die Castings—A. E. Keskulla and Junius D. Edwards, presented by Mr. Keskulla.

Finishing Magnesium Die Castings—H. W. Schmidt, presented by the author.

The Finishing of Zinc-Base Alloy Die Castings—E. A. Anderson, presented by the author.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by Sam Tour, chairman, and the following actions taken:

Accepted as Tentative:

Revisions of:

- Spec. for Aluminum Ingots for Remelting (B 24 - 39 T)
- Spec. for Aluminum Sheet and Plate (B 25 - 40 T)
- Spec. for Aluminum-Base Alloy Sand Castings (B 26 - 37 T)
- Spec. for Aluminum for Use in Iron and Steel Manufacture (B 37 - 39 T)
- Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 - 37 T)
- Spec. for Aluminum Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 - 40 T)
- Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 - 40 T)
- Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 40 T)
- Spec. for Aluminum Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 - 40 T)
- Spec. for Magnesium-Base Alloy Sheet (B 90 - 40 T)
- Spec. for Magnesium-Base Alloy Forgings (B 91 - 40 T)

Revisions of (*Continued*):
- Spec. for Magnesium-Base Alloys in Ingot Form for Sand Castings and Die Castings (B 93 - 40 T)
- Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 40 T)
- Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 38 T)
- Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 - 40 T)
- Spec. for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 - 38 T)

Adopted as Standard:

Revision of Spec. for Magnesium Ingot and Stick for Remelting (B 92 - 39)

The following appendices to the report were then presented as indicated:

A High Strength Aluminum Sand Casting Alloy not Requiring Solution Treatment and Quench, presented by D. Basch.

The Divider Method in Tension Testing of Magnesium-Base Alloys—H. W. Schmidt, presented by the author.

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented by E. M. Baker, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys

Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys

Revision of Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 40 T)

Editorial Change Accepted:

Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 - 40 T)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by Sam Tour, chairman, and the following action taken:

Accepted as Tentative:

Revision of Method of Salt Spray Testing of Non-Ferrous Metals (B 117 - 39 T)

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by C. H. Greenall, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Copper Sheet, Strip, and Plate, with the following editorial changes accepted by unanimous consent:

Section 1.—In the note at the end of this section omit item 3, renumbering the remaining items accordingly, and change the note to appear as Section 2 on "Basis of Purchase," renumbering the remaining sections accordingly.

Section 9.—Omit Paragraph (*b*) which reads as follows:

(*b*) Ninety-five per cent of the tension tests shall fall within the limits prescribed in Table I. In no case shall more than one test be permitted outside the limits, nor shall this vary more than 1000 psi. from the prescribed limits.

Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes

Spec. for Aluminum Bronze Rods, Bars, and Shapes

Spec. for Copper-Nickel-Zinc Alloy Rod and Wire, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 4 (*a*).—Change the first sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

Analysis shall be made of each lot of [10,000] *5000* lb. or fraction thereof.

Section 7.—Omit Paragraph (*b*) which reads as follows:

(*b*) Ninety-five per cent of the tension tests shall fall within the limits prescribed in Table II. In no case shall more than one test be permitted outside the limits, nor shall this vary more than 1000 psi. from the prescribed limits.

Spec. for Manganese Bronze Rods, Bars, and Shapes, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 8 (*a*).—Change the description of the test specimen to read as follows:

8. (*a*) All rods, bars, and shapes shall be pulled in full size when practicable. When a machined test specimen becomes necessary,

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[8] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee B-5, see p. 194.

enough metal may be removed from the gage section to meet the limitations of the testing machine, or the specimen may be machined to the form and dimension as shown in Fig. 9 of the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials. For rods, bars, and shapes up to $1\frac{1}{2}$ in. in diameter or thickness, the axis of the test specimen shall coincide with the central axis of the piece. For rods over $1\frac{1}{2}$ in. in diameter or thickness, the axis shall be located midway between the center and surface of the piece. The axis of tension test specimens for rods, bars, and shapes shall be parallel to the direction of rolling or drawing.

Spec. for Tin-Bronze and Leaded Tin-Bronze Sand Castings, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 1 (*b*).—Omit this section which reads as follows:

(*b*) Alloys 1A, 1B, and 2B shall not be used where castings are subjected to a temperature exceeding 500 F. (260 C.). Alloy 2A is suitable for applications involving temperatures of 500 F. (260 C.).

Table I.—Under alloy 2A change zinc from "4.00 per cent" to read "4.50 per cent"; under alloy 2B change copper from "88.50 per cent" to read "87.00 per cent"; omit from the table the column for nickel.

Table II.—Under alloys 2A and 2B change the maximum nickel content from "0.75 per cent" to read "1.00 per cent."

New Section.—Add the following section on certification:

16. *Certification*.—In the case of a product manufactured in advance and supplied for sale from stock by the manufacturer, jobber, or other dealer, the product may, upon request of the purchaser, be certified by the manufacturer as conforming to these specifications subject to the following procedures:

Not less than two tension tests, from different heats, and not less than one chemical analysis shall be made by the manufacturer from each day's melt. Records of the tension test results and chemical analysis shall be systematically made and maintained and shall be the basis for certification. In lieu of the manufacturer's certification and upon written request by the purchaser, these records may be examined by the purchaser or his accredited representative upon the manufacturer's premises.

Spec. for High-Leaded Tin Bronze Sand Castings

Spec. for Leaded Red Brass and Leaded Semi Red Brass Sand Castings, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 1 (*b*).—Omit this section which reads as follows:

(*b*) If castings are used under internal pressure conditions, alloy 4A is commonly used under hydrostatic pressure up to 350 psi. The other three alloys, namely, 4B, 5A, and 5B, may be used for correspondingly lower pressures following the sequence of the numerical designation.

Table II.—For all four alloys change the maximum nickel content from "0.50 per cent" to "1.00 per cent."

New Section.—Add a new section on certification to read as given above in the specifications for tin bronze.

Spec. for Leaded Yellow Brass Sand Castings for General Purposes

Spec. for High-Strength Yellow Brass and High-Strength Leaded Yellow Brass Sand Castings, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Table III.—Add the following requirements for yield strength:

Alloy	Yield Strength,[a] min., psi.
7A	20 000
8A	25 000
8B	40 000

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[a] Until such time as the committee shall have more information concerning each of these alloys, the yield strength shall be designated as the stress producing a permanent set of the material of 0.2 per cent as determined by the offset method.

Spec. for Aluminum-Bronze Sand Castings, with an editorial change, accepted by unanimous consent providing for the omission of the Brinell hardness requirements in Section 7.

Spec. for Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings.

In each of the above seven specifications for copper alloy castings editorial changes in the sections on chemical analysis and preparation of test specimens were accepted by unanimous consent.

Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing, with a change, accepted by unanimous consent, in the description of the test procedure to permit the use of hand-hammering in routine testing but not as a referee test in cases of dispute, subject to favorable letter ballot of Committee B-5.[8]

Mercurous Nitrate Test for Copper and Copper Alloys

Revisions of:

Spec. for Cartridge Brass, Sheet, Strip, and Disks (B 19 - 40 T)

Spec. for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 40 T), with the following correction accepted by unanimous consent:

Table II.—Under Alloy 9A change the requirement for copper from "85 per cent" to read "78 per cent"; under alloy 9B change the minimum aluminum content requirement from "8 per cent" to read "9 per cent"; also omit from this table footnote *d* which reads as follows: "Maximum impurities including iron but excluding manganese."

Spec. for Brass Sheet and Strip (B 36 - 40 T)

Spec. for Copper Pipe, Standard Sizes (B 42 - 39 T)

Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 39 T), with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 2.—Change the phrase "either of the two following types" to read "any one of the three following types," and add a new type as follows:

Type A.—Phosphorized copper, with low residual phosphorus.

Section 3.—Add the following requirements for chemical composition of Type A:

		Type A
Copper,[a] min., per cent		99.90
Phosphorus, per cent	min.	0.004
	max.	0.010

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[a] Silver counting as copper.

Spec. for Seamless Copper Tubes (B 75 - 40 T), the correction in the grain size in Table I from "0.40 mm." to "0.040 mm." to be made in the case of both soft annealed and light annealed material.

Spec. for Copper-Silicon Alloy Plate and Sheet (B 96 - 40 T), with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—CHEMICAL REQUIREMENTS.

	Type A	Type C
Copper, min., per cent	94.80	91.00
Silicon, per cent	2.75 to 3.50	2.00 to 3.00
One or more of the following elements may be present within the limits specified below, subject to limits of the specific alloys as given in the Appendix:		
Manganese, max., per cent	1.50	1.25
Zinc, max., per cent	1.50	4.50
Iron, max., per cent	1.60	0.80
Tin, max., per cent	0.75	....
Lead, max., per cent	0.05	0.05
Nickel, max., per cent	0.60	0.60
Sum of above elements, min., per cent	99.50	99.50

Section 8.—Change from its present form to read as follows:

8. *Weight Tolerances.*—The weight of each lot of five or more lot plates or sheets of the same specified dimensions when ordered to thickness shall not vary from the nominal by more than the amount prescribed in Table III (the accompanying Table II). The weight of any individual plate or sheet may vary from the nominal by not more than one third in excess of the tolerances prescribed in Table III. The tolerances of lots of less than five plates or sheets shall be governed by the tolerance of individual plates or sheets. For purposes of these specifications, types A and C shall be considered to weigh 0.308 lb. per cu. in.

TABLE II.—OVERWEIGHT TOLERANCES OF PLATES AND SHEET ORDERED TO THICKNESS.

Thickness, in.	Permissible Excess in Average Weight of Lots, Expressed in Percentage of Nominal Weights					
	48 in. and Under in Width	Over 48 to 60 in. in Width	Over 60 to 72 in. in Width	Over 72 to 96 in. in Width	Over 96 to 120 in. in Width	Over 120 to 132 in., incl. in width
1/8 to 3/16	6½	8	9	11	..	..
Over 3/16 to 1/4	6½	8	9	11	12	..
Over 1/4 to 5/16	6½	7¾	8¾	11	12	13
Over 5/16 to 3/8	6¼	7½	8½	11	12	13
Over 3/8 to 7/16	6	7¼	8¼	11	12	13
Over 7/16 to 1/2	6	7	8	10	11	12
Over 1/2 to 5/8	5¾	6½	7½	9	10	11
Over 5/8 to 3/4	5½	6	7	8	9	10
Over 3/4 to 1	5	5	6¼	7	8	9
Over 1 to 2	3½	4	5	6	7	8

Appendix.—Add as information in an Appendix at the end of these specifications a table giving detailed information on specific alloy compositions that are commercially available.

Spec. for Beryllium Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 - 40 T)

Spec. for Leaded Brass Sheet and Strip (B 121 - 39 T), with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 3.—In the table change the chemical composition requirements for alloy No. 5 to read as follows by the addition of the italicized figures and the omission of those in brackets:

Copper, per cent.........	[61.00 to 65.00]	*61.0 to 65.5*
Lead, per cent..	[1.75 to 2.25]	*1.5 to 2.5*
Iron, max., per cent	0.10	
Zinc, per cent..	remainder	

Spec. for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 39 T; ASA H7-1939)

Spec. for Gilding Metal Sheet and Strip (B 130 - 40 T)

Spec. for Brass Wire (B 134 - 40 T)

Spec. for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 - 40 T), with the following additional changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 5 (*a*).—Change from its present form to read as follows:

It is recommended that the tension test specimens shall be machined from coupons to the form and dimensions shown in Fig. 1, and shall not be chilled.

Section 7 (*b*).—Change from its present form to read as follows:

The sample for chemical analysis shall be taken by drilling or cutting the test coupon or sprue in such a manner as to be representative of the entire cross-section.

New Section.—Add a new section on certification to read as recommended above in the specifications for tin bronze.

Adopted as Standard:

Spec. for Brass Pipe, Standard Sizes (B 43 - 40 T)

Revisions of:

Spec. for Seamless Copper Boiler Tubes (B 13 - 40), with the following editorial change, accepted by unanimous consent:

Section 1.—Change the note from the form as preprinted to read as follows:

NOTE.—In the event copper boiler tubes for welding purposes are desired, deoxidized copper should be ordered to the Tentative Specifications for Seamless Copper Tubes (A.S.T.M. Designation: B 75) of the American Society for Testing Materials in so far as chemical composition requirements only are concerned. Other requirements shall be in accordance with these Standard Specifications B 13.

Spec. for Free-Cutting Brass Rod for Use in Screw Machines (B 16 - 29; ASA H8-1934), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Table II.—In the table as preprinted change the requirement for Half Hard Temper Material, 0.50 in. and less, from "57,000 psi." to read "60,000 psi."; the yield strength from "25,000 psi." to read "28,000 psi."; and the elongation in 2 in. from "10 per cent" to read "7 per cent"; also after the heading "Half Hard" in the first column, add "(Rounds, hexagons, and octagons)."

Add to this table the following requirements:

	Thickness, in.	Width, in.	Tensile Strength, min., psi.	Yield Strength, min., psi.	Elongation in 2 in., min., per cent
Half hard (rectangles and squares).......	1 and under	2 and under	50 000	25 000	10
	Over 1	Over 2	45 000	17 000	20

Spec. for Copper Water Tube (B 88 - 39; ASA H23.1-1939), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Sections 2 and 4 (*c*), *and Table II.*—Omit all references to type O tubing.

Table I.—Add the following as a footnote to the first item on light drawn temper: "Light drawn temper applies only to types K and L tubing."

Spec. for Sheet Copper-Silicon Alloy (B 97 - 40), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Table I.—Change to read as shown in the accompanying Table III.

Appendix.—Add as information in an Appendix at the end of these specifications a table giving detailed information on specific alloy compositions that are commercially available.

Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 40), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

TABLE III.—CHEMICAL REQUIREMENTS.

	Type A	Type B	Type C
Copper, min., per cent	94.80	96.00	91.00
Silicon, per cent	2.75 to 3.50	0.75 to 2.00[a]	2.00 to 3.00
One or more of the following elements may be present within the limits specified below, subject to limits of the specific alloys as given in the Appendix:			
Manganese, max., per cent	1.50	0.75	1.25
Zinc, max., per cent	1.50	1.50	4.50
Iron, max., per cent	1.60	0.80	0.80
Tin, max., per cent	0.75	1.60	....
Nickel, max., per cent	0.60	....	0.60
Lead, max., per cent	0.05	0.05	0.05
Sum of above elements, min., per cent	99.50	99.50	99.50

[a] An alloy containing as high as 2.60 per cent silicon is acceptable providing the sum of all elements, other than copper silicon, and iron does not exceed 0.30 per cent.

TABLE IV.—CHEMICAL REQUIREMENTS.

	Type A	Type B	Type C	Type D
Copper, min., per cent	94.80	96.00	91.00	94.00
Silicon, per cent	2.75 to 3.50	0.75 to 2.00[a]	2.00 to 3.00	2.75 to 3.50
Lead, per cent	....	....	....	0.20 to 0.80
One or more of the following elements may be present within the limits specified below subject to limits of the specific alloys as given in the Appendix:				
Manganese, max., per cent	1.50	0.75	1.25	1.50
Zinc, max., per cent	1.50	1.50	4.50	1.50
Iron, max., per cent	1.60	0.80	0.80	0.25
Tin, max., per cent	0.75	1.60	....	....
Lead, max., per cent	0.05	0.05	0.05	....
Sum of above elements, min., per cent	99.50	99.50	99.50	99.50

[a] An alloy containing as high as 2.60 per cent silicon is acceptable providing the sum of all elements, other than copper, silicon, and iron does not exceed 0.30 per cent.

Table I.—Change to read as shown in the accompanying Table IV.

Appendix.—Add as information in an Appendix at the end of these specifications a table giving detailed information on specific alloy compositions that are commercially available.

Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 40), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Table I.—Change to read as shown in the accompanying Table IV.

Appendix.—Add as information in an Appendix at the end of these specifications a table giving detailed information on specific alloy compositions that are commercially available.

Spec. for Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60 - 36), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 1 (c).—Omit this paragraph which reads as follows:

(*c*) It is recommended that this alloy shall not be used where castings are subjected to a temperature exceeding 500 F. (260 C.).

New Section.—Add a new section on certification as recommended above in the specifications for tin bronze.

Spec. for Steam or Valve Bronze Castings (B 61 - 40); with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 1.—Change from its present form: namely,

1. These specifications cover alloy castings, the alloy being a composition of copper, tin, lead, and zinc, commonly used as a high-grade steam metal or valve bronze, which is suitable for applications involving temperatures[a] of 500 F. (260 C).

to read as follows:

1. These specifications cover alloy castings, the alloy being a composition of copper, tin, lead, and zinc, commonly used as a high-grade steam metal or valve bronze.[a]

New Section.—Add a new section on certification as recommended above in the specifications for tin bronze.

Spec. for Composition Brass or Ounce Metal Castings (B 62 – 36), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 1.—Omit the second sentence, which reads as follows: "Castings of this alloy are commonly used in hydrostatic pressure up to 350 psi."

Add a new footnote to this section to read as follows:

The Rules for Construction of Unfired Pressure Vessels of the A.S.M.E. Boiler Construction Code (Par. U-20 (*f*)) require that composition or ounce metal conforming to these specifications shall be limited to temperatures of 406 F.

New Section.—Add a new section on certification as recommended above in the specifications for tin bronze.

Spec. for Copper Plates for Locomotive Fireboxes (B 11 – 33), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee B-5:[8]

Section 1.—Change from its present form to read as follows:

1. *Scope.*—These specifications cover four grades of copper plates for locomotive fireboxes as follows:

Grade 1, Tough pitch copper (nonarsenical)
Grade 2, Phosphorized copper (nonarsenical)
Grade 3, Tough pitch arsenical copper
Grade 4, Phosphorized arsenical copper

Section 3.—Change from its present form to read as follows:

3. *Chemical Composition.*—The copper shall conform to the following requirements as to chemical composition:

	Grade 1	Grade 2	Grade 3	Grade 4
Copper (plus silver), min., per cent...	99.90	99.90	99.40	99.40
Arsenic, per cent.......	...	...	0.15 to 0.50	0.15 to 0.50
Phosphorus, per cent .	...	0.015 to 0.040	...	0.015 to 0.040

Section 9.—Change to read as follows:

9. *Weight Tolerance.*—The weight of each lot of five or more plates of the same grade and the same specified dimensions when ordered to thickness shall not vary from the nominal by more than the amount given in Table I (the accompanying Table V). The weight of any individual plate may vary from the nominal by not more than one-third in excess of the tolerances given in Table I. The tolerance of lots of less than five plates shall be governed by the tolerance of individual plates. One cubic inch of rolled copper is assumed to weigh 0.323 lb.

The recommendation, appearing in the report as preprinted, for the withdrawal of the Standard Specifications B 60, B 61, and B 62 was omitted from the report by the committee, and in place thereof revisions in these three standards were presented for immediate adoption as listed above.

Withdrawal of Standards and Tentative Standards:

Spec. for Manganese-Bronze Ingots for Sand Castings (B 7 – 39), including existing tentative revision

Spec. for Manganese-Bronze Sand Castings (B 54 – 39), including existing tentative revision

Spec. for Aluminum Bronze Castings (B 59 – 39)

Spec. for Sand Castings of the Alloy: Copper 80 per cent, Tin 10 per cent, Lead 10 per cent (B 74 – 32 T), these specifications being superseded by the new Tentative Specifications for High-Leaded Tin Bronze Sand Castings

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[a] The temperature recommendation of the scope clause is conservative in view of known design practice. Establishment of definite limiting design stresses and of dimensional standards later may possibly permit some raising of the limiting temperature above that now recommended.

[a] The Rules for Construction of Unfired Pressure Vessels of the A.S.M.E. Boiler Construction Code (Par. U-20 (*f*)) require that bronze valves and steam metal conforming to these specifications shall be limited to temperatures of 550 F.

On the recommendation of the committee action was taken to incorporate in all of the specifications for copper alloy under the jurisdiction of Committee B-5 of a new section on "Basis of Purchase," as an aid to the purchaser in presenting all necessary details on a purchase order for material ordered according to the respective standards. This recommendation was accepted by unanimous consent, the change to be made editorially when the specifications are next printed.

Report presented by H. L. Curtis, chairman.

Committee D-9 on Electrical Insulating Materials:

Report presented, in the absence of the chairman, by Dean Harvey, vice-chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Round Phenolic Laminated Tubing for Radio Applications

Test for Punching Quality of Laminated Phenolic Sheets

Preconditioning Plastics and Electrical Insulating Materials for Testing, recommended jointly with Committee D-20 on Plastics

TABLE V.—HOT-ROLLED COPPER PLATES AND SHEETS, LOT WEIGHT TOLERANCES IN PERCENTAGE OF THEORETICAL WEIGHT, PLUS AND MINUS.

Thickness, in.	Weight Tolerances, plus and minus Percentage of Theoretical Weight				
	48 in. and Under	Over 48 to 60 in. in Width	Over 60 to 72 in. in Width	Over 72 to 90 in. in Width	Over 90 to 110 in. in Width
⅛ and under	8	9½	11	12½	14
Over ⅛ to 3/16	6½	8	9½	11	12½
Over 3/16 to ¼	6	7½	8½	9	10
Over ¼ to 5/16	5½	7	8	8½	9
Over 5/16 to ⅜	5	6	7	7½	8
Over ⅜ to 7/16	4½	5	6	7	7½
Over 7/16 to ½	4	4½	5½	6	6½
Over ½ to ⅝	3½	4½	5	5½	6
Over ⅝ to ¾	3	4	4½	5	5½
Over ¾ to 1	2¾	3½	4	4½	5
Over 1 to 1½	2½	3	3½	4	4½
Over 1½ to 2, incl.	2¼	2¾	3¼	3¾	4¼

The following papers, appended to the report, were then presented as indicated:

Influence of External Stresses on Tendency of Brass Wires to Stress-Corrosion Crack, as Indicated by the Mercurous Nitrate Test—Harry P. Croft, presented from manuscript by the author.

Mercury Cracking Test, Procedure and Control—H. Rosenthal and A. L. Jamieson, presented by Mr. Jamieson.

SIXTH SESSION—ELECTRICAL INSULATING MATERIALS, RUBBER

WEDNESDAY, JUNE 25, 2 P.M.

Session Chairman: J. J. Allen.

Sectional Committee C-59 on Electrical Insulating Materials:

Test for Volatile Matter Content of Vulcanized Fiber

Revisions of:

- Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 39 T)
- Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 40 T)
- Testing Glass Spool Insulators (D 550 – 39 T)

Adopted as Standard:

Testing Varnishes Used for Electrical Insulation (D 115 – 39 T)

Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 – 38 T), as revised

Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 – 38 T), as revised

Measuring Shrinkage from Mold Dimensions of

Molded Materials Used for Electrical Insulation (D 551 – 39 T)

Editorial Change Adopted:

Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 – 38; ASA C59.10–1941)

Published as Information Only:

Proposed Methods of Test for Product Uniformity of Phenolic Laminated Sheet[9]

Proposed Methods of Test for Sludge Formation in Mineral Transformer Oil

The proposed revision in the Tentative Method of Testing Pin-Type, Lime Glass Insulators (D 468 – 39 T), appearing in the report as preprinted, was withdrawn by the committee.

The following papers, appended to the report, were presented as indicated:

Measurements of Power Factor and Dielectric Constant at Ultra High Frequencies—K. G. Coutlee, R. F. Field, E. O. Hausmann, Thomas Hazen and Harry R. Meahl, presented by Mr. Meahl.

Report on Round-Robin Tests of Power Factor and Dielectric Constant for Glass—Philip A. Richards, presented by R. F. Field.

Committee D-11 on Rubber Products:

Report presented by O. M. Hayden, chairman, and the following actions taken:

Accepted As Tentative:

Testing Automotive Air Brake and Vacuum Brake Hose

Test for Tear Resistance of Vulcanized Rubber, with editorial corrections providing for changing the dimension "1.15 in." in Fig. 1 to read "1.14 in.," the ommission of the first half of the first sentence in Section 5, and the omission of Paragraph 7 (*b*).

Test for Compression Fatigue of Vulcanized Rubber

Revision of Methods of Testing Sponge Rubber Products (D 552 – 39 T)

Adopted as Standard:

Testing Flat Rubber Belting (D 378 – 40 T)

Tension Testing of Vulcanized Rubber (D 412 – 40 T), as revised

Test for Indentation of Rubber by Means of the Pusey and Jones Plastometer (D 531 – 39 T)

Testing Rubber Insulated Wire and Cable (D 470 – 40 T)

Spec. for Insulated Wire and Cable: Class AO 30 per cent Hevea Rubber Compound (D 27 – 40 T), as revised

Spec. for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 40 T), as revised

Spec. for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 40 T), as revised

Revisions of:

- Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 – 40)
- Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 – 40)
- Air Pressure Heat Test of Vulcanized Rubber (D 454 – 40)
- Sample Preparation for Physical Testing of Rubber Products (D 15 – 39)

The recommendation that the Tentative Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 – 39 T), as revised, be approved for reference to letter ballot of the Society for adoption as standard, failed to receive the necessary two-thirds vote of those voting at the session, the result of the vote being 2 affirmative, 9 negative.

Paper:

The following paper was presented:

A Method for the Testing of Adhesive Tape—William C. Geer and W. Burton Westcott, presented by Mr. Geer.

SEVENTH SESSION—COAL, COKE, GASEOUS FUELS

WEDNESDAY, JUNE 25, 2 P.M.

Session Co-Chairmen: R. B. Harper; Past-President A. C. Fieldner.

Committee D-5 on Coal and Coke:

Report presented by A. C. Fieldner, chairman, and the following actions taken:

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[9] See Report of Committee D-9, Editorial Note, p. 397.

Adopted as Standard:

Test for Index of Dustiness of Coal and Coke (D 547 - 39 T)

Withdrawal of Standard and Tentative Standard:

Definition of the Term Coke (D 121 - 26 T)
Spec. for Foundry Coke (D 17 - 16)

Editorial Change Adopted:

Laboratory Sampling and Analysis of Coal and Coke (D 271 - 40), elimination of note under Section 9

Committee D-3 on Gaseous Fuels:

Report presented by A. W. Gauger, chairman.

Papers:

The following papers were presented:

A Laboratory Test for the Ignitibility of Coal[10]—Ralph A. Sherman, J. M. Pilcher, and H. N. Ostborg, presented by Mr. Pilcher.

A Study of the Grindability of Coal and the Fineness of Pulverized Coal when Using the Lea-Nurse Air Permeability Method for Evaluating the Subsieve Fractions—J. B. Romer, presented from manuscript by the author.

Studies on the Measurement of Water Vapor in Gases—F. C. Todd and A. W. Gauger, presented by Mr. Gauger.

EIGHTH SESSION—ROAD AND PAVING MATERIALS, BITUMINOUS MATERIALS, PETROLEUM

WEDNESDAY, JUNE 25, 2 P.M.

Session Chairman: Past-President T. G. Delbridge.

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented, in the absence of the chairman, by E. H. Berger, vice-chairman, and the following actions taken:

Accepted as Tentative:

Revisions of:

- Spec. for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 37 T)
- Spec. for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 - 37 T)
- Spec. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 - 38 T)
- Spec. for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 - 37 T) with a change in the revision as preprinted to retain the original weights per roll of 62 lb. and 52 lb. for the two grades of roofing, respectively, rather than to change the present requirements to 61 lb. and 51 lb.
- Spec. for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 37 T)
- Spec. for Asphalt Cap Sheet Surfaced with Coarse Mineral Granules (D 371 - 37 T)

Adopted as Standard:

Spec. for Asphalt for Use in Constructing Built-Up Roof Coverings (D 312 - 39 T)
Spec. for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (D 450 - 38 T)
Spec. for Bituminous Grout for Use in Waterproofing Above Ground Level (D 170 - 39 T)
Spec. for Bituminous Grout for Use in Waterproofing Below Ground Level (D 171 - 39 T)
Spec. for Primer for Use with Asphalt in Dampproofing and Waterproofing (D 41 - 39 T)
Spec. for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (D 43 - 37 T)
Spec. for Asphalt Mastic for Use in Waterproofing (Asphalt Cement, Mineral Filler, Mineral Aggregate) (D 491 - 39 T), with editorial change
Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 37 T)
Testing Bituminous Mastics, Grouts, and Like Mixtures (D 147 - 39 T), with editorial change
Revision of Spec. for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 - 27)

Accepted as Information:

Proposed Spec. for Coal-Tar Pitch for Steep Built-up Roofs, Dampproofing, and Waterproofing, not preprinted, accepted by unanimous consent for publication as information as an appendix to the report of Committee D-8, subject to approval by Committee E-6 on Papers and Publications.

Committee D-4 on Road and Paving Materials:

Report presented by J. E. Myers, chairman, and the following actions taken:

Accepted as Tentative:

Tests for Modified Miscibility and Cement Mixing of Emulsified Asphalts
Revision of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine

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[10] Published in ASTM BULLETIN, No. 112, October, 1941, p. 23.

(C 131 – 39), with the following additional changes accepted by unanimous consent, this recommendation being presented jointly with Committee C-9 on Concrete and Concrete Aggregates:

Section 3 (b).—In the proposed revision of this section, delete the requirements for grading A, redesignating gradings B to E as A to D.

Section 4.—In the proposed revision of this section, eliminate from the table the requirements for grading A, redesignating gradings B to E as A to D; also delete the footnote to the table.

In explanation of the changes proposed in these specifications, append to the revision the following explanatory note:

EXPLANATORY NOTE.—The need for a wider range of gradings for use in the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39) has been met in part by the revisions presented this year by Committees C-9 and D-4. The new gradings C and D provide, in particular, for tests of the finer sizes used in concrete and in bituminous surface treatments. However, a further expansion in the range of gradings is needed to permit of tests of larger sizes of concrete aggregates and of aggregates such as are required for penetration macadam, water-bound macadam and other types of construction utilizing the coarser materials.

Gradings A and B, specified in the method as first adopted by the Society in 1937, require abrasive charges of 12 and 11 steel or cast iron spheres, approximately 1$\frac{7}{8}$ in. in diameter, respectively. Research leading to the development of the test method showed these charges and gradings to give substantially the same results for the same materials. Similarly, the new gradings C and D requiring 8 and 6 of the same spheres have been demonstrated to give approximately the same results. The data for these four gradings indicate the abrasive charges required for similar losses in tests of material from the same source to be a logarithmic function of the surface area of the test sample.

Based on the relationship referred to, the committees proposed at their spring meetings to include requirements for a coarser grading consisting of 2500 g. of 2½- to 2-in. material and 2500 g. of 2- to 1½-in. material tested with an abrasive charge of 15 of the steel or cast iron spheres. Subsequent tests showed the losses thus obtained to be, with minor exceptions, considerably lower than those found for the four gradings and charges specified in the proposed revised test method. Also tests made with more than 15 spheres as the abrasive charge failed to show proportionate increases in loss. Accordingly, the recommendation of the coarser grading was withdrawn at the June meeting.

In withdrawing that recommendation the committees instructed the subcommittees having the test method in charge to prepare the preceding statement in explanation of the basis for the revisions and to point to the need for further investigation.

Adopted as Standard:

Spec. for Preformed Expansion Joint Filler for Concrete (Nonextruding and Resilient Types) (D 544 – 39 T)

Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 – 39 T)

Test for Sieve Analysis of Mineral Filler (D 546 – 39 T)

This recommendation, not preprinted, for the adoption of the above three tentative standards was approved for reference to letter ballot of the Society subject to favorable letter ballot of Committee D-4,[11] these three tentative standards having been proposed for continuation as tentative in the report as preprinted.

It was announced that the committee had taken action to recommend to the Society through Committee E-10 on Standards subsequent to the annual meeting revisions in the following specifications and methods of test, which in the report as preprinted were proposed for continuation as tentative without revision.

Spec. for Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 37 T)

Methods of Sampling Bituminous Materials (D 140 – 39 T)

Spec. for Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 – 39 T)

Spec. for Crushed Stone for Bituminous Macadam Base and Surface Courses (D 192 – 39 T)

It was also announced that the following additional recommendations would be presented to the Society through Committee E-10 subsequent to the annual meeting:

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[11] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee D-4, see p. 368.

Proposed Tentative Specifications for Slow Setting Emulsified Asphalt for Fine Aggregate Mixes.
Proposed Tentative Specifications for Sodium Chloride
Proposed Tentative Volume Correction Table for Tar and Coal-Tar Pitch, prepared jointly with Committee D-8 on Bituminous Waterproofing and Roofing Materials
Tentative revision of Standard Method of Test for Ductility of Bituminous Materials (D 113 - 39)
Tentative revision of Standard Methods of Chemical Analysis of Calcium Chloride (D 345 - 34)

Committee D-18 on Soils for Engineering Purposes:

Report presented by C. A. Hogentogler, chairman, who stated that the Proposed Method of Test for Moisture Density Relations of Soil, appended to the report as information, would be acted on jointly by Committee D-18 and Committee D-4 on Road and Paving Materials before submission to the Society for publication as tentative. It was also announced that the Proposed Definitions of Terms and Symbols Relating to Soil Mechanics, appended to the report as information, may be submitted to Committee E-10 on Standards subsequent to the annual meeting, for publication as tentative.

Committee D-2 on Petroleum Products and Lubricants:

Report presented, in the absence of the chairman, by R. P. Anderson, secretary, and the following actions taken:

Accepted as Tentative:

Test for Aniline Point of Petroleum Products, with an editorial correction changing the tolerance for the 10-ml. pipette from "± 0.4 ml." to read "± 0.04 ml."

Test for Carbonizable Substances in Paraffin Wax, with the following change in the statement of scope accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[12]

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

1. This method of test is applicable to paraffin wax to ascertain whether it conforms to the standard of quality [prescribed by the U. S. Pharmacopoeia] ***required for pharmaceutical use.***

Test for Ignition Quality of Diesel Fuels

Test for Knock Characteristics of Aviation Fuels, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[12]

In the second paragraph of Footnote 2, change the second sentence to read as follows:

Based on tests of a variety of aviation fuels in current aircraft engines and on service experience this method satisfactorily rates such fuels for purposes of procurement and use under normal operating conditions.

Section 5 (q).—Change to read as follows by the addition of the italicized word and figure and the omission of the words in brackets:

(*q*) Humidity of Intake Air.—[The maximum permissible water content of the intake air shall be] ***25 to*** 50 grains ***of water vapor*** per pound of dry air.

Appendix I.—Change Paragraph A4 (*d*) to read as follows:

(*d*) *Valve Springs.*—Cadmium-plated. Pressure with valve open 104 ± 6 lb., with valve closed 83 ± 6 lb.

Change the first sentence in Paragraph A5 to read as follows by the addition of the italicized words and figure:

Necessary when the air in the room where the engine is operated contains ***less than 25 grains or*** more than 50 grains of water vapor per pound of dry air.

Change Paragraph A7 to read as follows:

A7. An evaporative cooling system with a water-cooled condenser coil above the coolant level. To maintain the specified temperature, a greater cooling capacity is required than for the apparatus specified in Tentative Method D 357.

Spec. for Aviation Gasoline, with a revision accepted by unanimous consent, subject to favorable letter ballot of Committee D-2[12] providing for changing the requirement for acidity of distillation residue for all grades from "neutral" to read "shall not be acid."

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[12] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee D-2, see p. 339.

Revisions of:

Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue (D 524 - 40 T)

Test for Gum Stability of Gasoline (D 525 - 40 T), with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[12]

Section 2 (d).—In the first sentence change "precision of not less than 50 psi." to read "precision of not more than 50 psi."

Section 3 (c).—Add the following as a note in connection with the second sentence:

NOTE.—It is permissible to add a higher boiling liquid such as ethylene glycol to the water in order to maintain the operating temperature of the bath at 212 ± 0.2 F. (100 ± 0.11 C.).

Section 5.—In the second sentence change "20 per cent" to read "10 per cent."

Test for Neutralization Number of Petroleum Products and Lubricants (D 188 - 27 T), with the following change accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[12]

Sections 1, 2 and 6.—Change the words "mineral acid" wherever they occur in these sections to read "strong acid."

Test for Saponification Number (D 94 - 39 T), with the following change accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[12]

Section 2 (a). Change to read as follows by the addition of the italicized words and the omission of those in brackets:

An Erlenmeyer flask of 300-ml. capacity *alkali-resistant,*[a] to which an adequate reflux or mushroom-type condenser is attached by means of a ground-glass joint. [The Erlenmeyer flask should preferably be made of Pyrex, Jena G-20, or equivalent alkali-resistant glass.]

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[a] *Pyrex and Kimble resistant glasses are suitable.*

Test for Sulfur in Petroleum Oils by Lamp Method (D 90 - 34 T), with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[12]

Section 3 (c).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

Dissolve 0.1 g. of methyl red in [100 ml. of ethyl alcohol (60 per cent)] *6 ml. of 0.0624 N solution of NaOH and dilute to 100 ml. with distilled water.*

Test for Tetraethyl Lead in Gasoline (D 526 - 39 T)

Test for Knock Characteristics of Motor Fuels (D 357 - 40), in the form of separate tentative methods, to replace immediately the existing standard; this recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee D-2.[12]

Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (D 565 - 40 T), with the following change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[12]

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

1. This method of test is applicable to white mineral oil (liquid petrolatum) to ascertain whether it conforms to the standard of quality [prescribed by the U. S. Pharmacopoeia] *required for pharmaceutical use.*

Adopted as Standard:

Test for Distillation of Plant Spray Oils (D 447 - 40 T), as revised

Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 40 T), as revised

Method for Calculating Viscosity Index (D 567 - 40 T)

Revisions of:

Test for Distillation of Crude Petroleum (D 285 - 36)

Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 39)

Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 - 38)

Published as Information Only:

Proposed Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity

Proposed Method of Test for Neutralization Number of Petroleum Products by Electrometric Titration, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[13]

Section 3.—Change the dimensions of the glass electrode in Paragraphs (*b*) and (*c*) to read as follows:

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[13] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 79, 58 members returned their ballots, of whom 52 voted affirmatively, 0 negatively.

Section	Present	Revised
3 (*b*)..	12.5 to 13.5 cm.	12.5 to 18.0 cm.
	1.0 to 1.4 cm.	0.8 to 1.4 cm.
	0.5 to 0.7 cm.	about 0.7 cm.
3 (*c*)..	12.5 to 13.5 cm.	12.5 to 18.0 cm.
	1.0 to 1.4 cm.	0.8 to 1.4 cm.
	2.0 to 2.5 cm.	0.8 to 2.5 cm.
	0.1-cm. hole	add "or holes"

Section 6 (*b*).—In the seventh sentence change "0.3 v." to read "0.03 v."; also make the same change in footnote 6.

Proposed Method of Test for Neutralization Number of New and Used Crankcase Oils

Proposed Method of Test for Potential Gum in Aviation Gasoline, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee D-2:[14]

Section 2 (*c*).—Change "not less than 50 psi." to read "not more than 50 psi."

Change Paragraph (*i*) to read as follows:

A sintered-glass Gooch crucible, fine porosity, of 25-ml. nominal capacity shall be used.

Section 3.—Omit this section which describes the preparation of the Gooch crucible, renumbering the remaining section accordingly.

Section 4 (*c*).—Change the second sentence to read as follows:

Maintain the temperature of the water at 212.0 ± 0.2 F. (100 ± 0.11 C.).

Section 4 (*e*).—Change to read as follows:

(*e*) Drain the oxidized sample from the glass liner into a beaker. To remove any insoluble gum, wash the interior of the liner and the catalyst together twice, using a fresh 10-ml. portion of the solvent (Note 6) for each washing. Add these washings to the oxidized sample, shake the mixture, and filter. Reserve the filtrate for the gum determination (Paragraphs (*f*) and (*g*)).

NOTE 10.—Avoid exposing the sample to daylight prior to and during this test. At the end of the test no visible precipitate shall be present in the glass liner.

Section 4 (*g*).—In the first sentence change the phrase "pour the filtrate into the beaker" to read "pour exactly one half of the filtrate plus washings into the beaker."

Section 4 (*h*).—In the first sentence add "per 100 ml." after "6.0 mg."

Diesel-Fuel-Oil Classification

Sectional Committee Z11 on Petroleum Products and Lubricants:

Report presented from manuscript, in the absence of the chairman, by the chair.

Papers:

The following papers were presented:

The Influence of Recovery Temperature on the Ductility of the Recovered Asphalt—Carl Bussow, presented by title.

Fundamental Significance of Oliensis Spot Test —Quantitative Tests for Homogeneity—G. L. Oliensis, presented by the author.

Performance Specifications for Greases—Robert C. Adams and Harrison E. Patten, presented by Mr. Adams.

NINTH SESSION—MARBURG LECTURE AND DUDLEY MEDAL AWARD

WEDNESDAY, JUNE 25, 4.15 P.M.

Session Chairman: President W. M. Barr.

In introducing the Sixteenth Edgar Marburg Lecturer, President Barr explained briefly the purpose of this annual memorial lecture, established 16 years ago as a means of honoring and perpetuating the memory of the first Secretary-Treasurer of the Society, Edgar Marburg, thus commemorating one who contributed so much to the A.S.T.M. during the years of its early development. It brings to the Society membership valuable expositions by authorities in the sciences and in engineering concerning important developments in the field of engineering materials.

He then presented Dr. H. L. Fisher, Director of Organic Research, U. S. Industrial Chemicals, Inc., and Air Reduction Co., Stamford, Conn., who had chosen as the subject of the lecture "Natural and Synthetic Rubbers." Doctor Fisher presented a comprehensive picture of the chemical and physical characteristics of crude rubber and the many synthetic rubber compositions, reviewing the contributions that research had made to the knowledge of the chemistry and physics of rubber, resulting in improved compounding and curing which have lengthened the life of the material and made possible applications, especially in engineering, which were previously impossible. The synthetic rubbers were reviewed at considerable length by the lecturer and he described the general structure of the many available compositions, their commercial production, properties, and applications. At the conclusion of the lecture, Doctor Fisher gave a detailed summary to indicate in what respects synthetic rubber compounds are superior to natural rubber compounds; and he also pointed out those properties in which natural rubber exhibits superiority over the synthetic compositions now available.

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[14] Committee D-2 reported results of the letter ballot vote as follows: Of a total voting membership of 79, 58 members returned their ballots, of whom 46 voted affirmatively, 1 negatively.

President Barr, in expressing appreciation to Doctor Fisher for his fine exposition on a subject of particular interest to the A.S.T.M., presented to him, on behalf of the Society, the Edgar Marburg Lecture Certificate.

President Barr then introduced R. W. Crum, chairman of the Dudley Medal Committee, who presented, as the recipient of the fifteenth award of the Charles B. Dudley Medal, Dr. C. W. MacGregor, Associate Professor of Applied Mechanics, Massachusetts Institute of Technology, for his paper on "The Tension Test," presented at the 1940 Annual Meeting of the Society. Mr. Crum explained that the medal is awarded annually by the Society to the author of a technical paper considered to be of outstanding merit, and constituting an original contribution on research in engineering materials. In thus recognizing meritorious contributions in the materials field, the medal commemorates the name of the first President of the Society, Charles B. Dudley.

On behalf of and as the representative of the Society, President Barr then made the fifteenth award of the Charles B. Dudley Medal to Doctor MacGregor.

In expressing appreciation of the honor conferred upon him by this award Doctor MacGregor made the following remarks:

> It gives me a great deal of pleasure to receive this Dudley Medal. I wish to express my deep appreciation of this high honor which this Medal confers and at the same time extend my sincere thanks to the American Society for Testing Materials and the Dudley Medal Committee. This Medal, I am sure, will be a source of great inspiration to me in the future, and I only hope that, whatever future results we may get, I will obtain some results which will justify somewhat, or at least in some measure, the confidence which you have expressed in me today. Again, I thank you.

The session was then followed by an informal reception arranged by the Chicago Committee during which opportunity was provided for the members, guests, and their ladies to meet the new and retiring officers of the Society.

The evening was designated as Officers' Night in the Exhibit of Testing Apparatus and Related Equipment and many of the past and present officers attended.

TENTH SESSION—STEEL, EFFECT OF TEMPERATURE

THURSDAY, JUNE 26, 9.30 A.M.

Session Co-Chairmen: President W. M. Barr; P. G. McVetty

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman.

Joint Research Committee on Effect of Temperature on the Properties of Metals:

Report presented by N. L. Mochel, chairman, and the following action taken:

Adopted as Standard:

Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 38 T), as revised

Mr. Mochel then called upon **F. B. Foley** to present from manuscript the report on Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures, prepared by H. W. Gillet, which is to be issued as a separate publication.

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Low-Alloy Structural Steel, with the following change accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Section 4 (*a*).—Change the requirement as preprinted for maximum manganese content in ladle analysis and check analysis, respectively, from "1.00" and "1.05" to read "1.25" and "1.30" per cent.

Section 7.—Add a new Paragraph (*d*) to read as follows:

(*d*) For material under $\frac{5}{16}$ in. in thickness or diameter, a deduction from the percentage of elongation in 8 in. specified in Paragraph (*a*) of 1.25 per cent shall be made for each decrease of $\frac{1}{32}$ in. of the specified thickness or diameter below $\frac{5}{16}$ in.

Spec. for Carbon-Steel and Alloy-Steel Ring and Disk Forgings with the following changes accepted by unanimous consent subject to favorable letter ballot of Committee A-1:[15]

Section 1 (*b*).—Substitute for Paragraph (*b*) as preprinted, the following new Paragraphs (*b*) and (*c*) relettering the subsequent paragraph accordingly:

(*b*) A ring-shaped or a disk-shaped forging whose length as measured on its longitudinal axis is less than its diameter or major transverse dimension is considered a ring or a disk within the meaning of these specifications.

(*c*) It is recognized that certain hollow cylindrical forgings whose length as measured on their longitudinal axes are more than their diameters or major transverse dimensions may be considered rings within the scope of these specifications if they have been forged in a manner similar to that of rings, namely, expanded in diameter on a mandrel.

Section 2 (*b*).—Renumber as Section 3 on Manufacture and change from its form as preprinted to read as follows:

3. The forgings shall receive their hot mechanical work under a press or a hammer, or in a mill, each of ample power to work the metal uniformly throughout its section.

Section 4.—Omit this section as preprinted, renumbering the subsequent sections accordingly.

Section 9.—Change the second sentence from its form as preprinted to read as follows:

Drillings for analysis may be taken from the forging or from a full-size prolongation or extension, at any point midway between the inner and outer surfaces of the prolongation left on one end of a ring forging or at any point midway between the upper and lower surfaces of the prolongation left on the rim of a disk or ring forging, or turnings may be taken from a test specimen.

Section 10,—Change Paragraphs (*b*) and (*c*) from their form as preprinted to read as follows:

(*b*) The size classification as shown in Table II shall be determined for a ring forging by its thickness or width, whichever is the lesser dimension. The thickness shall be measured by the difference between the outside and inside radii. The width shall be measured parallel to the axis of the ring.

For a disk forging the size classification shall be determined by its thickness as measured parallel to its axis.

(*c*) The prolongation or extension for test specimens shall be added in the location which will not increase the lesser dimension of the section, and hence not change the size classification of the forging.

In the first sentence of Paragraph (*d*) change the crosshead speed from "$\frac{1}{8}$ in. per min." to read "$\frac{1}{2}$ in. per min."

Section 12.—Substitute for Paragraphs (*a*) and (*b*) as preprinted, the following new paragraphs (*a*) to (*f*) relettering the subsequent paragraphs accordingly:

(*a*) Tension test specimens and, if specified, bend test specimens for forgings weighing 5000 lb. or more each, shall be taken from extensions of the forgings; or when approved by the purchaser, the test specimens may be taken from the forgings with a hollow drill.

(*b*) For forgings weighing less than 5000 lb. each, subject to the requirements of Section 13, test specimens shall be taken as specified in Paragraphs (*c*), (*d*), (*e*), and (*f*).

(*c*) Unless otherwise specified, coupons for test specimens may be prepared from a small test ingot taken during the pouring of the steel, or from a portion of the billet used to make the forgings. The coupons shall receive approximately the same amount of mechanical work and the same heat treatment as the forgings they represent.

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[15] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee A-1, see p. 71.

(*d*) When specified, the test specimens shall be taken from a prolongation or extension of the forging, except as prescribed in Paragraph (*f*).

(*e*) When tests are to be taken from prolongations or extensions of the forgings, unless otherwise specified, at least 20 per cent of the forgings from each melt shall have such prolongations or extensions which shall be made on the outside diameter or edge of a disk, or on the width or thickness of a ring (see Section 10 (*c*)).

(*f*) Test specimens may be taken from a forging if the manufacturer elects to submit a forging to represent each test lot in lieu of extensions; or when approved by the purchaser, the test specimens may be taken from the forgings with a hollow drill.

Section 13.—Omit Paragraph (*c*) and (*d*) as preprinted, relettering the subsequent paragraphs accordingly, and add new Paragraphs (*c*) and (*e*) to read as follows:

(*c*) For heat-treated forgings weighing less than 5000 lb. each, one tension test shall be made from each size classification represented in each melt in each annealing or normalizing charge; or from each size classification in each melt in each normalizing or quenching charge represented in each tempering charge.

(*e*) When bend tests are specified by the purchaser the number shall be the same as for tension tests specified in Paragraphs (*a*), (*b*), and (*c*).

Spec. for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings, comprising a tentative revision of the Standard Specifications A 17 - 29, with the following changes accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Section 2.—Change from its form as preprinted to read as follows:

2. These specifications cover standard forging quality material. Requirements other than, or more restrictive than those prescribed herein will constitute a special requirement quality, details of which shall be agreed upon by the manufacturer and the purchaser.

Table I.—Make the following changes in the chemical requirements as preprinted:

Type	Element	Change from	To Read
Steel 1021	Manganese, per cent	0.60 to 0.90	0.70 to 1.00
Steel 1030	Carbon, per cent	0.27 to 0.33	0.28 to 0.34
Steel 3316[a]	Carbon, per cent	0.13 to 0.18	0.14 to 0.19
Steel 4140	Carbon, per cent	0.37 to 0.44	0.38 to 0.45
	Manganese, per cent	0.60 to 0.90	0.70 to 1.00
Steel 4150	Molybdenum, per cent	0.15 to 0.25	0.15 to 0.27
Steel 4320	Carbon, per cent	0.18 to 0.23	0.17 to 0.22
Steel 4336	Carbon, per cent	0.32 to 0.38	0.34 to 0.40
Steel 4341	Carbon, per cent	0.37 to 0.44	0.38 to 0.45
Steel 4620	Carbon, per cent	0.18 to 0.23	0.17 to 0.22
Steel 4820	Manganese, per cent	0.30 to 0.60	0.40 to 0.70

[a] This type number formerly read "3315."

Section 11.—Omit this section as preprinted renumbering the subsequent sections accordingly.

Spec. for Heat-Treated Wrought Steel Wheels, with the following editorial changes to bring Section 12 in line with the corresponding specifications of the Association of American Railroads:

Section 12 (*a*).—Add the words "by class," so that the sentence will read: "The wheels shall be mated by class as to one-half tape size and shipped in pairs."

Section 12 (*b*).—Delete this section.

Spec. for Light Gage Structural Quality Flat Hot-Rolled Carbon Steel 0.2499 and 0.1874 in. to 0.0478 in. Inclusive, in Thickness with the following change accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Table I.—Change the specified gage weight of "2.000 and heavier" to read "1.875 and heavier," and omit the permissible variations from specified gage weight for weights 1.874 and lighter.

Spec. for Light Gage Structural Quality Flat Rolled Carbon Steel 0.0477 in. to 0.0225 in. Inclusive, in Thickness

Revisions of:

Spec. for Alloy-Steel Forgings for Locomotives and Cars (A 238 - 40 T)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 40 T)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 40 T); with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Sections 2 (d), 12 and Paragraphs S5 and S7.—Change the mandatory grain size requirement to make this optional with the purchaser, adding at the appropriate portions of these sections and paragraphs the phrase: "When requested by the purchaser and so stated in the order."

Section 2 (*e*).—Add the phrase: "When the steel melting practice specified in Section 2 (*d*) is required," in order to bring this section into agreement with the new policy of optional grain size requirements.

Section 20 (*c*), *Items* (*2*) *and* (*3*).—The proposed changes in these sections as preprinted in the report were withdrawn, thus continuing the present requirements concerning surface conditions.

Spec. for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 40 T); with the following additional changes accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Table I.—Change the carbon content of grade P 5c (4 to 6 per cent chromium-molybdenum stabilized with titanium or columbium) from "0.10 or as specified 0.15 max.," to read "0.12 max." per cent; change the titanium content from its present value of "4 to 6 times carbon" to read "min., per cent, 4 times the actual carbon; max. 0.70" per cent.

Section 19 (*c*), *Items* (*2*) *and* (*3*).—The proposed changes in these sections as preprinted in the report were withdrawn, thus continuing the present requirements concerning surface conditions.

Spec. for Steel for Bridges and Buildings (A 7 - 39)

Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 40)

Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 - 39), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Section 1.—Change the second sentence to read as follows by the omission of the words and figures in brackets:

The pipe is intended for conveying liquid, gas, or vapor [at temperatures below 450 F.]; and only grade A is adapted for flanging and bending.

Section 14 (*a*).—Change the requirement on the pipe bevel from its present figure of "45 deg." to read "37.5 deg." Throughout this section change the figure "45 deg." to read "37.5 deg."

Spec. for Electric-Resistance-Welded Steel Pipe (A 135 - 34), with the following additional change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Sections 1 and 14 (*a*).—Make the same change in these sections as recommended above in Specifications A 139 - 39.

Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 30 in. and Over) (A 134 - 39), with the following change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Section 1.—Change the second sentence to read as follows by the omission of the words and figures in brackets:

The pipe is intended for conveying liquid, gas, or vapor [at temperatures below 450 F.]

Spec. for Forged or Rolled Steel Pipe Flanges for General Service (A 181 - 37)

Spec. for Welded and Seamless Steel Pipe (A 53 - 40)

Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 40)

Spec. for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 - 40)

Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 40), with the following change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Table I.—Under grade T 16 (chromium-molybdenum-titanium) change the carbon content from "0.10 max." to read "0.12 max."; also change the titanium content from "0.35 to 0.60" to read "min., per cent, 4 times actual carbon; max. 0.70 per cent."

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 - 40 T), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Section 1.—Change the last two sentences in Note 2 to read as follows:

Grade WCB is applicable to the design standards formulated by the Sectional Committee on Standardization of Pipe Flanges and Fittings, under the procedure of the American Standards Association, as given in the American Standard for Steel Pipe Flanges and Flanged Fittings (ASA No.: B16*e* - 1939). The Sectional Committee is now developing pressure ratings for the other grade of material covered in these specifications.

Section 16.—Delete the Note, and insert a new paragraph (*b*) to read as follows:

(*b*) Finished products shall be marked in accordance with the Standard Marking System for Valves, Fittings, Flanges, and Unions (No.

SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.

Spec. for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 40 T), with the following changes, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Section 1.—Change the last two sentences in Note 2 to read as follows:

Grades WC1 and WC4 are applicable to the design standards formulated by the Sectional Committee on Standardization of Pipe Flanges and Fittings, under the procedure of the American Standards Association, as given in the American Standard for Steel Pipe Flanges and Flanged Fittings (ASA No.: B16*e* - 1939). The Sectional Committee is now developing pressure ratings for the other grades of material covered in these specifications.

Section 17.—Make the same change in this section as recommended above in Specifications A 216 - 40 T.

Following similar action as mentioned above in the case of Specifications A 206 - 40 T and A 158 - 40 T, the tentative revision of Standard Specifications A 106 - 40 appearing in the report as preprinted was withdrawn by the committee.

Adopted as Standard:

Spec. for Hot-Worked High-Carbon Steel Tie Plates

Spec. for High-Strength Structural Rivet Steel (A 195 - 39 T)

Spec. for Hard-Drawn Steel Spring Wire (A 227 - 39 T)

Spec. for Steel Music Spring Wire (A 228 - 39 T)

Spec. for Oil-Tempered Steel Spring Wire (A 229 - 39 T)

Spec. for Carbon-Steel Valve Spring Quality Wire (A 230 - 39 T)

Spec. for Chromium-Vanadium Steel Spring Wire (A 231 - 39 T)

Spec. for Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 - 39 T)

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 - 39 T), as revised.

Revisions of:

Spec. for Wrought Steel Wheels for Electric Railway Service (A 25 - 36), with a correction in Section 8 on diameter of hub, as preprinted, by addition of the following sentence:

The thickness of the hub wall in any wheel shall not vary more than ⅜ in. at any two points equidistant from face of hub.

Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 40), with the following additional change, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Table I.—Change the requirement for manganese content of grade A (seamless) from "0.30 to 0.60" to read "0.30 to 0.90," per cent.

Spec. for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 - 40), with the following additional change, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

New Grade.—Add a new grade of chromium-molybdenum steel, designated C 12, making the necessary changes in Tables I and II and editorially in other sections of the specification, the requirements for chemical composition and tensile properties being as follows:

Chemical Requirements:	Grade C 12
Carbon, per cent	0.3 max.
Manganese, per cent	1.00 max.
Phosphorus, per cent	0.05 max.
Sulfur, per cent	0.06 max.
Silicon, per cent	1.00 max.
Chromium, per cent	8.00 to 10.00
Molybdenum, per cent	1.10 to 1.50

Physical Requirements:	Grade C 12
Tensile strength, min., psi	90 000
Yield point, min., psi	60 000
Elongation in 2 in., min., per cent	18
Reduction of area, min., per cent	30

Section 17.—Delete the Note and insert a new Paragraph (*b*) reading as follows:

(*b*) Finished products shall be marked in accordance with the Standard Marking System for Valves, Fittings, Flanges, and Unions (No. SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.

Spec. for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 - 40), with the following change, not preprinted, accepted by unanimous consent, subject to favorable letter ballot of Committee A-1:[15]

Section 16.—Make the same change in this section as recommended above in Section 17 of Specifications A 157 - 40.

Sectional Committee B36 on Standarization of Dimensions and Materials of Wrought Iron and Wrought Steel Pipe and Tubing:

Progress report presented in the absence of the chairman, by Sabin Crocker, secretary.

Committee A-9 on Ferro-Alloys:

Report presented, in the absence of the chairman, by the Assistant Secretary, and the following actions taken:

Adopted as Standard:

Spec. for Spiegeleisen (A 98 - 39 T)

The Tentative Specifications for Ferrochromium (A 101 - 39 T) were continued as tentative.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

Report presented by Jerome Strauss, chairman, and the following actions taken:

Adopted as Standard:

Recommended Practice for Conducting Plant Corrosion Tests (A 224 - 39 T), as revised

Spec. for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 - 40 T)

Revisions of:

- Spec. for Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (A 176 - 39)
- Spec. for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip and Plate (A 167 - 39)

The revision proposed for immediate adoption in the Standard Specifications for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 - 39), which appeared in the report as preprinted, was withdrawn by the committee subject to favorable letter ballot of Committee A-10.[16]

Papers:

The following papers were presented:

The Effect of Carbide Spheroidization upon the Creep Strength of Carbon-Molybdenum Steel —S. H. Weaver, presented from manuscript by the author.

The Fabrication of Carbon-Molybdenum Piping for High-Temperature Service—R. W. Emerson, presented by the author.

Compression and Tension Tests for Structural Alloys—Bruce Johnston and Francis Opila, presented from manuscript by Mr. Opila.

The Stress-Strain Characteristics of Cold-Rolled Austenitic Stainless Steels in Compression as Determined by the Cylinder Test Method—Russell Franks and W. O. Binder, presented by Mr. Franks.

Addition of Bismuth for Producing Free-Machining Stainless Steels—H. Pray, R. S. Peoples, and F. W. Fink, presented by Mr. Pray.

ELEVENTH SESSION—TIMBER, TEXTILES, PAINT, SOAP, PAPER, FIRE TESTS OF MATERIALS

THURSDAY, JUNE 26, 9.30 A.M.

Session Co-Chairmen: Past-President Hermann von Schrenk; R. D. Bonney.

Committee D-7 on Timber:

Report presented by Hermann von Schrenk, chairman, and the following actions taken:

Adopted as Standard:

Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions (D 453 - 39 T)

Revision of Definitions of Term Relating to Timber Preservatives (D 324 - 33), term water-gas tar

Committee C-5 on Fire Tests of Materials and Construction:

Report presented, in the absence of the chairman, by H. M. Robinson, secretary, and the following actions taken:

Accepted as Tentative:

Test for Fire-Retardant Properties of Wood

Adopted as Standard:

Fire Tests of Door Assemblies (C 152 - 40 T)

Revision of Spec. for Fire Tests of Building Construction and Materials (C 19 - 33; ASA A2-1934), as revised

Committee D-10 on Shipping Containers:

Report presented, in the absence of the chairman, by the chair.

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[16] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee A-10, see p. 129.

Committee D-6 on Paper and Paper Products:

Report presented by Lewis S. Reid, chairman, and the following actions taken:

Adopted as Standard:

Test for Bulking Thickness of Paper (D 527 - 39 T)
Test for Machine Direction of Paper (D 528 - 39 T)
Test for Water-Soluble Acidity or Alkalinity of Paper (D 548 - 39 T)
Test for Resin in Paper (D 549 - 39 T)

It was announced that the committee plans to present to the Society through Committee E-10 on Standards, subsequent to the annual meeting, the following six methods for publication as tentative, the first three having appeared in the preprinted report as information:

Test for Folding Endurance of Paper
Test for Basis Weight of Paper
Test for Thickness of Paper and Paper Products
Method for Conditioning Paperboard and Fiberboard and Paperboard Containers for Testing Boxes for Testing
Method for Compression Testing of Corrugated and Solid Fiber Boxes
Test for Moisture in Paper

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by H. E. Smith, chairman. The recommendations on standards appearing in the report were presented by M. Rea Paul, secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Oiticica Oil (Permanently Liquid)
Spec. for Liquid Paint Driers with the following change accepted by unanimous consent:

Section 2 (*a*).—In the table of color numbers omit the requirement for cobalt liquid standard of 39.3.

Spec. for Diatomaceous Silica Pigment, with a correction in the requirement for loss on ignition changing the maximum value as preprinted from "10.0 per cent" to read "1.0 per cent."

Spec. for Barium Sulfate Pigments with the following changes accepted by unanimous consent:

Section 2 (*a*).—Change the text from its form as preprinted to read as follows:

(*a*) The pigment shall consist of barium sulfate ($BaSO_4$) without any admixture of other materials in the case of blanc fixe, and without any admixture of other materials not naturally occurring in the barite ore in the case of barytes, and which conform to the following requirements.

In the Table of requirements for composition and properties add the following new requirement for free silica:

	Barytes	Blanc Fixe
Free silica (quartz, clays, or other foreign materials), max., per cent.	2.0	2.0

Spec. for Mica Pigment
Spec. for Aluminum Silicate Pigment
Spec. for Magnesium Silicate Pigment with the following change accepted by unanimous consent:

Section 2 (*a*).—Add a new requirement to the table to read "Magnesium oxide, per cent . . . 20.0 to 32.0."

Spec. for Lead Titanate
Spec. for Dibutylphthalate
Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service
Method for Evaluating the Degree of Resistance to Rusting on Painted Iron or Steel Surfaces
Test for Color of Orange Shellac

Revisions of:

Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 40 T) with the following changes accepted by unanimous consent:

Omission from the report as preprinted of the definitions of the terms "dry" and "drying;" also change the definition of the term "gallon" from its form as preprinted to read as follows:

Gallon, United States.—A volume of 231 cu. in. at 15.5 C. (60 F.).

Gallon, Imperial.—A volume of 231 cu. in. at 15.5 C. (60 F.).

Sampling and Testing Aluminum Powder and Paste (D 480 - 38 T)
Testing Liquid Driers (D 564 - 40 T)

Test for Specular Gloss of Paint Finishes (D 523 - 39 T) with the following additional changes accepted by unanimous consent:

Section 3 (a).—At the end of this section add a new footnote 5 to read as follows:

Constructions differing from that specified may be employed if such constructions are proved to give identical results.

Sections 5 (a) and 7 (a).—Change the standard value for specular gloss from "one" to read "one thousand."

Section 5 (b).—Change the last sentence from its form as preprinted to read as follows:

Surfaces of liquids may also be used[a] as working standards.

Section 6 (c).—Add a new footnote 10 at the end of this paragraph to read as follows:

For determining the specular gloss of specimens similar in reflecting characteristics to an available standard, larger apertures yield equally accurate results which are often more precise.

Adopted as Standard:

Spec. for Raw Soybean Oil (D 124 - 37 T), as revised
Spec. for Shellac Varnishes (D 360 - 39 T)
Spec. for Ultramarine Blue (D 262 - 40 T), as revised
Spec. for Titanated Lithopone (D 477 - 40 T), to be added to Standard Spec. D 477
Spec. for Carbon Black (D 561 - 40 T), as revised
Test for Consistency of Enamel Type Paints (D 562 - 40 T)
Test for Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus (D 522 - 39 T)

Revisions of:

Spec. for Leaded Zinc Oxide (D 80 - 40)
Sampling paragraph, affecting 24 pigment specifications: D 79 - 39, D 80 - 40, D 81 - 38, D 82 - 38, D 83 - 39, D 84 - 40, D 85 - 40, D 209 - 30, D 210 - 30, D 211 - 40, D 212 - 40, D 213 - 40, D 261 - 40, D 263 - 40, D 264 - 40, D 266 - 39, D 267 - 31, D 405 - 38, D 474 - 39, D 475 - 40, D 476 - 39, D 477 - 39, D 478 - 40, D 520 - 40
Spec. for Raw Tung Oil (D 12 - 33)
Spec. for Perilla Oil, Raw or Refined (D 125 - 23)
Spec. for Orange Shellac (D 237 - 33)
Testing Drying Oils (D 555 - 39)
Chemical Analysis of White Linseed Oil Paints (D 215 - 37)
Sampling and Testing Lacquer Solvents and Diluents (D 268 - 33), incorporation of Tentative Methods D 268 - 39 T

Committee D-17 on Naval Stores:

Report presented, in the absence of the chairman, by the chair.

Committee D-13 on Textile Materials:

Report presented by H. J. Ball, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Medium-Weight Cotton Corduroy Fabrics
Spec. for Fire-Retardant Properties of Treated Textile Fabrics
Test for Evaluating Compounds Designed to Increase the Resistance of Textiles to Insect Pests, with the following changes, accepted by unanimous consent:

Section 3 (b).—Designate this paragraph as a separate Section 4 on "Duration," renumbering the remaining sections accordingly, also the references in Table I.

Testing Asbestos Tubular Sleeving
Quantitative Analysis of Textiles, with Section 12 (*a*) changed to read as follows:

12. (*a*) A clean fiber residue obtained in Section 10 which contains cotton and wool may be used. However, it is recommended that a sample of the original textile be used since acetate rayon, silk, regenerated cellulose rayon, and cotton are all removed completely in this method. The sample shall include a complete pattern of such materials as plaids. If a 4 to 5-g. sample is taken, the sample may be cut diagonally to make two test specimens. Dry the specimen, weighing approximately 2 g., to constant weight at 105 to 110 C. in accordance with Section 4 (*c*) to obtain the weight of the dried specimen, weight *B*.

Delete Paragraph (*c*) and reletter the present Paragraph (*d*) as (*c*).

Test for Commercial Weight of Continuous Filament Rayon Yarns (D 258 - 41 T)
Test for Commercial Weight of Spun Rayon Yarns and Threads (D 507 - 41 T)

Revisions of:

Spec. for Single-Ply Bleached Cotton Broadcloth (D 504 - 38 T)
Testing and Tolerances for Woven Glass Fabrics (D 579 - 40 T)

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[a] D. G. Moore and R. S. Hunter, "Use of Liquid Surfaces as Standards of Specular Gloss," *J. Am. Ceramic Soc.*, Vol. 24, p. 167 (1941).

Testing and Tolerances for Woven Glass Tapes (D 580 – 40 T)
Testing Rayon Staple (D 540 – 39 T)
Test for Resistance of Textile Fabrics and Yarns to Moths (D 582 – 40 T)
Test for Shrinkage of Grease Wool (Laboratory Scale Operations) (D 584 – 40 T)
Identification of Fibers in Textiles and Quantitative Analysis of Textiles (D 276 – 37 T)
Spec. for Textile Testing Machines (D 76 – 40)
Spec. and Test for Fineness of Wool Tops (D 472 – 41)
Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 38)
Testing and Tolerances for Cotton Yarns (D 180 – 40)
Testing Pile Floor Covering (D 418 – 40)
Definitions of Terms Relating to Textile Materials (D 123 – 40)

Adopted as Standard:

Testing and Tolerances for Single Jute Yarn (D 541 – 39 T), as revised
Spec. and Test for Fineness of Wool Tops (D 472 – 40 T)

The recommendation for adoption as standard of the Tentative Definitions of Terms Relating to Textile Materials (D 123 – 40 T), as revised, was discussed on behalf of Committee E-8 on Nomenclature and Definitions. Since certain of the definitions included in the recommendation were of interest to other standing committees and in order to provide opportunity for review of the definitions before formal adoption by the Society, Committee E-8 had taken action requesting this recommendation be deferred for a year in order that any desirable changes may be considered before formal adoption as standard by the Society. It was brought out, however, that the definitions in question represented only a few that were being combined with the present standard definitions, and this recommendation was accordingly approved for reference to letter ballot of the Society with the understanding that Committee E-8 would review all of the definitions within the coming year.

Revisions of:

Testing and Tolerances for Cotton Yarns (D 180 – 40)
Testing and Tolerances for Tubular Sleeving and Braids (D 354 – 36; ASA L13-1941)
Test for Fastness of Dyed or Printed Cotton Fabrics to Laundering or Domestic Washing (D 435 – 37), with the following addition accepted by unanimous consent:

Section 6 (*c*).—Add the following sentence at the end of this section:

The operations of rinsing and drying shall be performed with as little interval between each operation as possible.

Test for Fastness of Colored Textile Fabrics to Light (D 506 – 39)
Spec. for Textile Testing Machines (D 76 – 40)
Spec. and Test for Asbestos Tape for Electrical Purposes (D 315 – 37)
Spec. and Test for Asbestos Roving for Electrical Purposes (D 375 – 37)

Withdrawal of Standards:

Spec. and Test for Asbestos Yarns (D 299 – 37)
Spec. and Test for Cuban (Jute) Raw Sugar Bags (D 275 – 33)

Committee D-12 on Soaps and Other Detergents:

Report presented, in the absence of the chairman, by C. C. Zeigler, and the following actions taken:

Accepted as Tentative:

Spec. for Olive Oil Chip Soap (Type A, Pure; Type B, Blended)
Test for Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution-Volumetric Method)

Revisions of:

Spec. for Palm Oil Chip Soap (D 536 – 39 T)
Spec. for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 – 40 T)
Sampling and Chemical Analysis of Special Detergents (D 501 – 40 T)
Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 40 T)

Adopted as Standard:

Spec. for Built Soap, Powdered (D 533 – 39 T)
Spec. for Soap Powder (Alkaline Soap Powder) (D 534 – 39 T)
Spec. for Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 – 40 T)

Spec. for Sodium Metasilicate (D 537 - 39 T)
Spec. for Sodium Sesquisilicate (D 594 - 40 T)
Chemical Analysis of Sulfonated and Sulfated Oils (D 500 - 38 T)
Revision of Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 39)

Papers:

The following papers were presented:

Tests of Glued Laminated Wood Beams Impregnated with Creosote—William A. Oliver, presented by the author.

The Effect of Rate of Loading on Tensile Strength of Cord and Yarn—M. Castricum and A. N. Benson, presented by Mr. Benson.

TWELFTH SESSION—METHODS OF TESTING

THURSDAY, JUNE 26, 2 P.M.

Session Co-Chairmen: Lincoln T. Work; Vice-President G. E. F. Lundell.

Committee E-8 on Nomenclature and Definitions:

Report presented from manuscript by Cloyd M. Chapman, chairman.

It was announced that the committee had approved for adoption as standard the tentative revision in the Standard Definitions, with Units and Symbols, Relating to Magnetic Testing (A 127 - 39) proposed by Committee A-6 on Magnetic Properties, the revised definitions of the terms gypsum lath, gypsum sheathing board, and gypsum wall board as proposed by Committee C-11 on Gypsum, and the definition of water-gas tar recommended by Committee D-7 on Timber.

It was also announced that the committee had reviewed the Tentative Definitions Relating to Textile Materials (D 123 - 40 T) proposed for adoption as standard by Committee D-13 on Textile Materials, and the tentative definitions of the terms abrasion, corrosion, erosion, and slagging, recommended for adoption as standard by Committee C-8 on Refractories, and had taken action to request these committees to defer formal adoption of these definitions for a year so that they may be reviewed by Committee E-8 and in order that any desirable changes may be discussed with the committees before adoption as standard by the Society.

Committee E-2 on Spectrographic Analysis:

Report presented by H. V. Churchill, chairman.

Committee E-3 on Chemical Analysis of Metals:

Report presented from manuscript by G. E. F. Lundell, chairman.

Committee E-1 on Methods of Testing:

Report presented by W. H. Fulweiler, chairman. The following resolution, presented by Mr. Fulweiler on behalf on Committee E-1 was, on motion, adopted:

WHEREAS, The American Society for Testing Materials is devoted to the standardization of methods of testing engineering materials and accordingly is concerned with the development and use of the scientific apparatus required in the conduct of these tests; and

WHEREAS, The Society includes among its membership the testing engineers, metallurgists, and chemists whose work is absolutely essential to our National Defense Program, and

WHEREAS, The Society through its Technical Committee on Laboratory Apparatus is cognizant of the very serious situation that confronts the manufacturers of scientific apparatus in that they now find it impossible to secure the necessary raw materials, many of which come under the classification of critical materials; and

WHEREAS, The manufacture of this essential scientific apparatus requires only a very small quantity of such critical materials; therefore, be it

Resolved, That the American Society for Testing Materials assembled in Chicago, Illinois, at its Forty-fourth Annual Meeting urges the U. S. Government in the interest of protecting our National Defense Program to take immediate action through some blanket form of priority to the end that manufacturers of scientific apparatus may procure the necessary supply of critical materials.

Resolved, That this action is urged due to the fact that the apparatus must be made in quantity, and the procuring of individual priorities for material for each piece of testing apparatus would involve complications and expense far beyond the actual cost of the apparatus.

Resolved, That this action be transmitted to the appropriate department of the U. S. Government with the request that it be given prompt consideration in view of its urgent nature.

Mr. Fulweiler then called upon H. F. Moore to describe the activities of Technical Committee I on Mechanical Testing; on E. C. Bingham to report on the work of Technical Committee II on Consistency, Plasticity, and Related Properties; on L. T. Work to summarize the activities of Technical Committee III on Particle Size and Shape; and on R. F. Passano to report on the activities of Technical Committee IX on Interpretation and Presentation of Data.

Mr. Fulweiler also called attention to the following two papers appended to the report:

Effect of Specimen Shape and Size on Measured Values of Tensile Properties—G. F. Jenks.
Effect of Rate of Strain on Yield Strength, Tensile Strength, Elongation, and Reduction of Area in Tension Tests—P. G. Jones and H. F. Moore.

The paper by J. J. Moran on "Methods of Testing Volumetric Glassware," to be appended to the report, was presented from manuscript by the author.

Paper:

The following paper was presented:

Statistical Theory of the Effect of Dimensions and of Method of Loading upon the Modulus of Rupture of Beams—John Tucker, Jr., presented by D. E. Parsons.

The address on "The Electron Microscope and Its Uses," by R. Bowling Barnes, Director, Physics Division, American Cyanamid Co., was then presented. In the presentation Mr. Barnes showed on the screen a number of interesting photographs illustrating the use and possible applications of the electron microscope.

THIRTEENTH SESSION—WATER

THURSDAY, JUNE 26, 2 P.M.

Session Co-Chairmen: President W. M. Barr; R. E. Hall.

Committee D-19 on Water for Industrial Uses:

Report presented, in the absence of the chairman, by F. N. Speller, vice-chairman, and the following actions taken:

Adopted as Standard:

Sampling Plant or Confined Waters for Industrial Uses (D 510 - 40 T)
Reporting Results of Analysis of Industrial Waters (D 596 - 40 T)
Determination of Hydroxide Ion in Industrial Waters (D 514 - 38 T)

Joint Research Committee on Boiler Feedwater Studies:

Report[17] presented from manuscript by C. H. Fellows, chairman.

Symposium on Problems and Practices in Determining Steam Purity by Conductivity Methods:

The symposium was introduced by R. E. Hall, and the following papers were then presented:

The Sampling of Steam and Boiler Water—A. R. Belyea and A. H. Moody, presented by Mr. Moody.
Experimental Methods of Determining Conductivity Corrections for Dissolved Gases in Steam Condensate—S. F. Whirl and W. A. Lower, presented by Mr. Whirl.
Calculation of Corrections to Conductivity Measurements for Dissolved Gases—D. S. McKinney, presented by the author.
The Degasification of Steam Samples for Conductivity Tests—P. B. Place, presented by the author.

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[17] Published in ASTM BULLETIN, No. 111, August, 1941, p. 56.

A New Type of Conductivity Apparatus for Use with Boiler Waters and Steam Samples—A. R. Mumford, presented from manuscript by the author.

Conductivity Cells and Electrical Measuring Instruments—C. E. Kaufman, presented from manuscript by the author.

FOURTEENTH SESSION—FATIGUE OF METALS, CORROSION OF IRON AND STEEL

THURSDAY, JUNE 26, 8 P.M.

Session Co-Chairmen: C. E. Heussner; Past-President H. F. Moore.

Committee A-5 on Corrosion of Iron and Steel:

Report presented, in the absence of the chairman, by R. F. Passano, and the following actions taken:

Adopted as Standard:

Spec. for Zinc-Coated Steel Wire Strand (Galvanized and "Extra Galvanized") (A 122 - 39 T), to supersede Standard Spec. A 122 - 33

Spec. for Zinc-Coated Steel Wire Strand (Class B and Class C Coatings) (A 218 - 39 T)

Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A 239 - 40 T), to supersede Standard Method A 191 - 38 and Tentative Method A 208 - 38 T

Research Committee on Fatigue of Metals:

Report presented by H. F. Moore, chairman, who also summarized the report on the investigation on "The Effect of Type of Testing Machine on Fatigue Test Results" appearing in the Appendix.

Papers:

The following papers were presented:

An Accelerated Atmospheric Corrosion Test—H. Pray and J. L. Gregg, presented from manuscript by Mr. Gregg.

An Equation Representing the Rate of Development of Rust on Galvanized Iron Sheets as Estimated by the A.S.T.M. Test—J. B. Austin, presented by C. D. Hocker.

The Effect of Shot Blasting and Its Bearing on Fatigue—J. M. Lessells and W. M. Murray, presented by Mr. Murray.

Fatigue Comparison of 7-in. Diameter Solid and Tubular Axles—O. J. Horger and T. V. Buckwalter, presented by Mr. Horger.

Pitting and Its Effect on the Fatigue Limit of Steels Corroded Under Various Conditions—D. J. McAdam, Jr., and G. W. Geil, presented by Mr. McAdam.

Testing Material in the Resonance Range—R. K. Bernhard, presented by the author.

A New High-Temperature Fatigue Machine—W. P. Welch and W. A. Wilson, presented by Mr. Welch.

FIFTEENTH SESSION—CEMENTITIOUS MATERIALS, BUILDING MATERIALS

THURSDAY, JUNE 26, 8 P.M.

Session Co-Chairmen: F. H. Jackson; Past-President H. H. Morgan.

Committee C-16 on Thermal Insulating Materials:

Report presented, in the absence of the chairman, by E. T. Cope, secretary, and the following actions taken:

Accepted as Tentative:

Test for Compressive Strength and Flexural Strength of Preformed Block Type Thermal Insulating Materials

Sampling and Preparation of Specimens for Testing of Thermal Insulating Cements

Test for Bulk Density of Thermal Insulating Cements

Test for Covering Capacity and Volume Change upon Drying of Thermal Insulating Cements

Test for Thickness and Density of Blanket Type Thermal Insulating Materials

Definitions of Terms Relating to Thermal Insulating Materials: Preformed thermal insulating block, thermal insulating cement

Sectional Committee A42 on Specifications for Plastering:

Informal progress report presented, in the absence of the chairman, by J. W. McBurney, secretary.

Committee C-7 on Lime:

Report presented by Nathan C. Rockwood, chairman.

Committee C-11 on Gypsum:

Report presented, in the absence of the chairman, by H. J. Schweim, secretary, and the following actions taken:

Accepted as Tentative:

Revision of Definitions of Terms Relating to Gypsum (C 11 - 40 T)

Revision of Methods of Testing Gypsum and Gypsum Products (C 26 - 40); this recommendation, which appeared in the report for immediate adoption, was modified and action taken to publish the revision for a year as tentative, with the understanding that it would be submitted to letter ballot of the Society for adoption as standard in 1942.

Adopted as Standard:

Revisions of:

- Spec. for Gypsum (C 22 - 25)
- Spec. for Gypsum Partition Tile or Block (C 52 - 33)
- Definitions of Terms Relating to Gypsum (C 11 - 40 T), terms gypsum lath, gypsum sheathing board, and gypsum wall board.

Publication as Information Only:

Proposed Method for Determination of Calcium Sulfate in Gypsum by the Ammonium Acetate Method, prepared by L. S. Wells and W. F. Clarke, and modified by B. E. Kester, was presented by Mr. Kester and accepted for publication as an Appendix to the report.

Committee C-1 on Cement:

Report presented by P. H. Bates, chairman, and the following actions taken:

Accepted as Tentative:

Revision of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 - 38 T)

Adopted as Standard:

Spec. for Portland Cement (C 150 - 40 T) as revised, to replace Standard Spec. for Portland Cement (C 9 - 38), and for High-Early-Strength Portland Cement (C 74 - 39)

Committee C-12 on Mortars for Unit Masonry

Report presented by J. W. McBurney, chairman, and the following action taken:

Accepted as Tentative:

Spec. for Mortar for Reinforced Brick Masonry, with the following changes approved by unanimous consent, subject to favorable letter ballot of Committee C-12:[18]

Section 4.—Change to read as follows by the addition of the italicized figure and the omission of that in brackets:

4. Mortar and grout, after suction for 60 sec. shall have a flow greater than [65] *70* per cent of the flow immediately after mixing.

Section 9 (a).—Change item (*1*) to read as follows by the addition of the italicized figure and the omission of that in brackets:

(*1*) One part of portland cement [0.12] *0.21* parts of hydrated lime (or its equivalent as lime putty), and not more than 3 parts of dry aggregate by weight.

Committee C-14 on Glass and Glass Products:

Report presented, in the absence of the chairman, by G. E. F. Lundell, and the following action taken:

Accepted as Tentative:

Definition of the Term Glass

It was announced that Methods of Chemical Analysis of Soda-Lime Glass had been submitted to letter ballot vote in the committee and would be presented to the Society for publication as tentative through Committee E-10 on Standards subsequent to the annual meeting.

Committee C-18 on Natural Building Stones:

Report presented, in the absence of the chairman, by H. A. Stacy, and the following actions taken:

Withdrawal of Tentative Standards:

Test for Compressive Strength of Natural Building Stone (C 98 - 30 T)

Sampling Natural Building Stone and Preparation of Sample for Testing (C 101 - 32 T)

Tension Testing of Natural Building Stone (C 103 - 32 T)

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[18] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee C-12, see p. 296.

Definitions of Terms Relating to Natural Building Stones (C 104 - 31 T)

Committee C-15 on Manufactured Masonry Units:

Report presented by D. E. Parsons, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Vitrified Clay Filter Block for Trickling Filters

Adopted as Standard:

Revisions of:

- Spec. for Structural Clay Load-Bearing Wall Tile (C 34 - 39)
- Spec. for Structural Clay Non-Load-Bearing Tile (C 56 - 39)
- Methods of Sampling and Testing Brick (C 67 - 39)

Committee C-8 on Refractories:

Report presented, in the absence of the chairman, by H. C. Cross, and the following actions taken:

Accepted as Tentative:

Revision of Classification of Insulating Block and Insulating Fire Brick (C 155 - 40 T)

Adopted as Standard:

Testing Fireclay Refractories Under Load Test at High Temperatures (C 16 - 39 T)
Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (C 20 - 39 T)
Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change) (C 93 - 39 T)
Test for Size and Bulk Density of Refractory Brick (C 134 - 39 T)
Chemical Analysis of Chrome Ore and Chrome Refractories (C 18 - 40 T)
Classification of Fireclay Refractories (C 27 - 40 T)
Test for Warpage of Refractory Brick and Tile (C 154 - 40 T)
Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 40 T)
Spec. for Refractories for Moderate Duty Stationary Boiler Service (C 153 - 40 T)
Spec. for Refractories for Incinerators (C 106 - 40 T)
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 40 T)
Revision of Spec. for Ground Fire Clay (C 105 - 36)
Revision of Symbols for Heat Transmission (C 108 - 39)

The recommendation for adoption as standard of the terms abrasion, corrosion, erosion, and slagging appearing in the Tentative Definitions of Terms Relating to Refractories (C 71 - 40 T) was discussed on behalf of Committee E-8 on Nomenclature and Definitions. In view of the general nature of these definitions which are of interest to other standing committees action had been taken by Committee E-8 requesting that this recommendation be deferred for a year in order that these definitions may be reviewed and any desirable changes discussed with Committee C-8 before formal adoption as standard by the Society. The recommendation for adoption failed of approval and was referred back to the committee.

Papers:

The following papers were presented:

Properties of Limes and Lime-Mortars—Howard R. Staley, presented from manuscript by the author.
An Improved Hydrometer Method for Use in Fineness Determinations—Alexander Klein, presented from manuscript by R. E. Davis.
The Effect of Fine Aggregate on the Durability of Mortars—C. A. Hughes and K. A. Anderson, presented from manuscript by H. F. Gonnerman.
Effect of Freezing Temperature on Freezing-and-Thawing Tests of Brick—Joseph C. Richmond and John W. McBurney, presented by Mr. McBurney.

SIXTEENTH SESSION—CONCRETE AND CONCRETE AGGREGATES SANFORD E. THOMPSON AWARD

FRIDAY, JUNE 27, 9.30 A.M.

Session Co-Chairmen: H. J. Gilkey; R. W. Crum.

Committee C-13 on Concrete Pipe:

Report presented by Theodore Doll, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Concrete Sewer Pipe (C 14 - 40 T), to replace Standard Specifications C 14 - 35

Spec. for Reinforced Concrete Sewer Pipe (C 75 - 40 T), as revised, to replace Standard Specifications C 75 - 35

Spec. for Reinforced Concrete Culvert Pipe (C 76 - 40 T), as revised, to replace Standard Specifications C 76 - 37

Papers for Revised Report on Significance of Tests of Concrete and Concrete Aggregates:

The papers for the revised Report on Significance of Tests of Concrete and Concrete Aggregates were introduced by the chairman, R. W. Crum, following which the papers were presented as follows:

Significance of Tests for Size, Shape, Surface Texture, and Grading of Aggregates—H. J. Gilkey, presented by the author.

Density and Unit Weight of Concrete—M. A. Swayze, presented by the author.

Mineral Composition of Aggregates—Herbert F. Kriege, presented from manuscript by the author.

Significance of Permeability and Absorption of Concrete—M. O. Withey, presented by the author.

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by F. H. Jackson, chairman, and the following actions taken:

Accepted as Tentative:

Revisions of:

Spec. for Ready Mixed Concrete (C 94 - 39), with the following change in Section 2, accepted by unanimous consent, subject to favorable letter ballot of Committee C-9:[19]

Section 2.—Change the definition of ready mixed concrete to read as follows by the addition of the italicized words:

The term ready mixed concrete is used to describe mixed concrete (either central mixed or transit mixed) delivered *in a plastic state* to the site of the work ready for use.

Section 14 (b).—To correct an error in this section as preprinted, change to read as follows by the addition of the italicized words and the omission of those in brackets:

Each sample of concrete [for strength tests] shall consist of not less than one cubic foot made up of portions [of] *obtained from* at least three representative parts of the batch.

Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 40), with changes as given earlier being presented jointly with Committee D-4 on Road and Paving Materials, was accepted by unanimous consent, subject to favorable letter ballot of Committee C-9.[20]

Spec. for Concrete Aggregates (C 33 - 40)

Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (C 87 - 39)

A recommendation, presented on the floor by C. E. Wuerpel, that the Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 39 T) be revised as follows was accepted by unanimous vote, subject to favorable letter ballot of Committee C-9,[21] as required by Society regulations:

Section 6 (a).—Change the first sentence to read as follows by the addition of the italicized words and figure, and the omission of the figure in brackets:

The samples shall be immersed in the prepared solution of sodium sulfate or magnesium sulfate for [18] *at least 16* hr. in such a manner that the solution covers them to a depth of at least ½ in.

In the last sentence change the words "18-hr. period" to read "immersion period."

In Paragraph (*b*) omit "18 hr." from the first sentence.

Mr. Jackson then announced that the Sanford E. Thompson Award, established by Committee C-9 in honor of the first chairman of the committee, and for the purpose of stimulating research and encouraging investigations that extend the knowledge of concrete and concrete aggregates, had been made to W. T. Thomson, Assistant Professor, Department of Applied Mechanics, Kansas State College, for his paper on "A Method of Measuring Thermal Diffusivity and Conductivity of Stone and Concrete," presented at the 1940 annual meeting. In the absence of the recipient, C. H. Scholer accepted from Mr. Jackson the certificate of the Sanford E. Thompson Award, expressing appreciation on behalf of Mr. Thomson for the honor conferred.

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[19] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee C-9, see p. 288.

[20] See p. 17.

[21] Results of the letter ballot vote as reported by Committee C-9 are as follows: Of a total voting membership of 74, 61 members returned their ballots, of whom 52 voted affirmatively, 0 negatively.

The chairman then recognized Cloyd M. Chapman who presented the following resolution, which was unanimously adopted for transmission to the Chicago District Committee:

> *Resolved,* That the Society record its appreciation to the Chicago Committee for having sponsored the 1941 annual meeting and for its untiring and eminently fruitful efforts in the development and execution of plans that contributed so greatly to the success of the meeting, and that this resolution be spread upon the minutes of the meeting, and that a copy be sent to the committee.

Papers:

The following papers were presented:

The Effect of Capping Methods and End Conditions Before Capping upon the Compressive Strength of Concrete Test Cylinders—G. E. Troxell, presented by R. E. Davis.

Concrete Under Sustained Working Loads; Evidence that Shrinkage Dominates Time Yield—G. A. Maney, presented by the author.

A Device for Studying the Workability of Concrete—T. C. Powers and E. M. Wiler, presented by Mr. Powers.

Discussion of Dynamic Methods of Testing Concrete with Suggestions for Standardization—Leonard Obert and Wilbur I. Duvall, presented from manuscript by Mr. Obert.

SEVENTEENTH SESSION—IRON

FRIDAY, JUNE 27, 9.30 A.M.

Session Chairman: H. C. Aufderhaar.

Committee A-6 on Magnetic Properties:

Report presented by Thomas Spooner, chairman, and the following actions taken:

Adopted as Standard:

Test for Measuring Interlamination Resistance and Lamination Factor of Iron and Steel (A 34 - 40 T)

Revision of Test for Magnetic Properties of Iron and Steel (A 34 - 39), with a correction in the description of the high-H permeameter in Section 7 (*d*) as preprinted, changing "500 oersteds" to read "5000 oersteds."

Revision of Definition of Terms, with Units and Symbols, Relating to Magnetic Testing (A 127 - 39)

Sectional Committee A21 on Specifications for Cast Iron Pipe and Fittings:

Informal progress report presented, in the absence of the chairman, by J. T. MacKenzie.

Committee A-7 on Malleable-Iron Castings:

The chairman recognized J. J. Kanter, who, on behalf of Committee A-7 on Malleable Iron Castings, stated that the report as preprinted had been withdrawn by the committee in order that further consideration might be given to the preliminary proposal in the report concerning the use of the terms "yield point" and "yield strength" in connection with the specifications for malleable iron castings under the jurisdiction of the committee.

Committee A-3 on Cast Iron:

Report presented, in the absence of the chairman, by J. T. MacKenzie, vice-chairman, and the following actions taken:

Accepted as Tentative:

Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron, with an editorial change to transfer the note at the end of Section 1 to appear at the beginning of the recommended practice.

Adopted as Standard:

Spec. for Cast-Iron Pit-Cast Pipe for Water and Other Liquids (A 44 – 39 T)

Revision of Spec. for Automotive Gray-Iron Castings (A 159 – 40).

Revision of Spec. for Gray-Iron Castings (A 48 – 36), with the following change, not preprinted, accepted by unanimous consent subject to favorable letter ballot of Committee A-3:[22]

Table I.—For Class No. 60 iron add a minimum breaking load requirement of 12,500 lb. in the transverse or flexure test on 2.0 in. diameter bar (Type C) tested on 24-in. supports.

Papers:

The following papers were presented:

Measurement of Core Loss and A. C. Permeability with the 25-cm. Epstein Frame—S. L. Burgwin, presented by the author.

The Strain Hardening of Gray Cast Iron—John Sanford Peck, presented by title.

Fatigue and Static Load Tests of a High-Strength Cast Iron at Elevated Temperatures—W. Leighton Collins and James O. Smith, presented by Mr. Collins.

Young's Modulus of Elasticity and Some Related Properties of Graphitic Materials—H. A. Schwartz and C. H. Junge, presented by Mr. Junge.

There being no further business the chairman then declared the Forty-fourth Annual Meeting adjourned *sine die.*

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[22] Results of the letter ballot on this recommendation, indicating its approval, are given in the report of Committee A-3, see p. 86.

SPEED, SPECIFICATIONS, AND SAFETY

ANNUAL ADDRESS BY THE PRESIDENT

W. M. BARR[1]

June 24, 1941

In preparing this address my Scotch instinct and my sympathetic nature prevailed. I have therefore reduced the cost of printing and given due consideration to the sensitive nature of this audience.

Not long ago it was said that the public was no longer interested in locomotives and in rail transportation, but recently published statistics indicate that 62 per cent of the nation's freight tonnage is still handled by the railroads. Airways carry less than 1 per cent, highways 9 per cent, pipelines 12 per cent, while inland shipping carries 17 per cent. Omitting the inland waterways, the railroads carry approximately 75 per cent of all overland freight tonnage.

This causes one to wonder what could be done in the present National Emergency without rail transportation. Quoting Mr. L. K. Sillcox in a recent article:

> "Whatever developments may arise in the future, railways will remain for many years the bulwark of our inland transportation needs."

It seems fitting at this time to repeat a stanza from James Russell Lowell which I quoted soon after taking office as President of this Society:

> New occasions teach new duties;
> Time makes ancient good uncouth;
> They must upward still, and onward
> Who would keep abreast of truth.
>
> *From "The Present Crisis" (1844).*

In the days when dobbin was the chief motive power of the country and the "one hoss shay" or similar vehicles constituted the rolling stock, transportation was at speeds which gave the public no concern. The designer was not required to present specifications for materials used, and the factor of safety was not a part of his mathematical training.

The story of "Darius Green and His Flying Machine" was looked upon as the fanciful and ridiculous imaginings of a humorist and the impossibility of the undertaking was so obvious that the reader invariably reacted to the story with a laugh as the author intended. Were the writer of this jingle alive today he would probably say "I told you so." With the development of the steam locomotive, and speeds of thirty to forty miles per hour, the traveler began to fear for his safety, and the engineers to give more consideration to strength of materials, calculation of stresses, and due observance of reasonable factors of safety.

When three days were required to go by rail from Chicago to Denver it was considered a long and hazardous journey. Now when the business man takes sixteen hours for the same trip he sleeps as comfortably as at home and is safer than in his home town for the same period of time. Nor does he consider making the same trip by air in less than six hours a particularly serious undertaking.

What factors have entered into this development of speeds beyond the wildest dreams of our grandsires?

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[1] Chief Chemical and Metallurgical Engineer, Union Pacific Railroad Co., Omaha, Nebr.

When the steam locomotive first exceeded 150,000 lb. in weight and had steam pressure of 150 lb. it was considered a large power unit. Some of these locomotives are still in operation either on small branch lines or as switching engines. The large passenger locomotive today weighs 440,000 lb., has steam pressure of 300 lb. and moves a heavy train at 90 miles per hour. The steam pressure of these locomotives is double that used but a few years earlier. Large freight locomotives today weigh more than half a million pounds, and some have been built with a total weight of locomotive and tender of more than a million pounds and having a tractive effort of 135,000 lb. and attaining speeds of seventy miles per hour when operating under load. The Diesel-electric power unit has a speed as high as one hundred twenty miles per hour, and the electric locomotive as much.

Yet the number of accidents on the railroads today is smaller than forty years ago. Irresponsible automobile drivers who fail to use care at grade crossings have caused many accidents, but they are neither train passengers nor railroad employees. Unfortunately, specifications cannot be written to cover drivers of cars nor can their alcoholic content be controlled, nor can they be forced to obey rules. Therefore the only solution of this safety problem lies not in specifications or speed restrictions but in grade separation. This involves large expenditures and will take time to accomplish, but much has been done through cooperation of the Federal Government and the state governments, with the railroads. The time will come when highway traffic will be completely separated from all main line railroads.

What has been necessary to maintain the excellent safety record of the railroads in the face of constantly increasing speeds, heavier loads, longer trains, larger locomotives with increased boiler pressures and the introduction of the high-speed Diesel-electric power units? The answer lies in improved designs, new and higher-strength materials, better maintenance of both roadway and equipment, constant watchfulness by supervisory forces, and last but not least a continuous safety campaign by an organized force.

The wooden passenger car with its fire hazard and eggshell body is a thing of the past, having been replaced by the sturdy steel construction of the modern and safe passenger car. But with larger cars and larger locomotives required for successful handling of greater tonnage and the more rapid movement of perishable freight, and the public demanding faster transportation of passengers to meet the requirements of the increasing tempo of the business world, new problems were introduced.

To meet the increased stresses imposed by both weight and speed, the designer used larger sections in structural members and thicker plates in boiler shells and car bodies until both cars and locomotives were approaching the practical limits of weight and wheel and axle loads. The weight of rail sections had gradually increased to make possible the safe movement of greater loads at increased speeds.

With the development of the automobile came the first use of alloy steels to obtain more strength with less weight. The research metallurgist introduced new elements into steel making and studied the possibilities of special heat treatments and further refinements in the production of the higher grades of steel. Much was accomplished by such studies, but little was done in the application of these research results to the large sections required for locomotives and railway cars until about 1920. It was then discovered that new metallurgical problems were introduced by the greater mass of metal required for heavy forgings.

But these problems were solved and the locomotive designer who had worked with heavy forgings having a minimum yield point of 40,000 psi. had at his command heat-treated alloy steels with minimum yield point of 70,000 psi. or more. Thus he was able to reduce the weight of axles and reciprocating parts and at the same time increase the factors of safety, which was desirable because of increasing speed.

Many heavy castings have been given improved tensile properties with reduction in weight by the addition of alloys. Then came the introduction of the high tensile strength low-alloy steels in car construction. This brought about a reduction in freight car weights of twenty-five per cent and gave the railroads much lighter passenger cars of more pleasing design.

The introduction of stainless steel having high physical properties and resistance to corrosion is an outstanding contribution to light-weight passenger car construction. The researches of the metallurgists brought out higher tensile strength aluminum alloys, much lighter in weight than steel. These are now extensively used in passenger car construction for high-speed trains, with notable weight reduction.

The development of high-strength alloy steels has made it possible for designers to reduce the weight of Diesel engines which, together with electric-motor drive, makes one of the most practical power units for high-speed rail transportation. The ability of this type of power unit to make runs of twenty-five hundred miles or more without servicing has made possible marked reductions in the time schedules.

The wheels on high-speed trains introduced a serious problem. The wheel that had been satisfactory for many years was no longer safe nor was its service acceptable. Metallurgical research with studies in heat treatment made higher specification limits possible and resulted in the production of wheels that are safe for high-speed service and in a measure meet the more exacting requirements of this service. This is still an open field for study and improvement.

Although rails were rolled with larger sections to meet the requirements imposed by greater power and increased speeds, rail failures continued and in many cases increased. The ever-present transverse fissure became more frequent and therefore more dangerous. This resulted in more expensive maintenance and required greater watchfulness to avoid accidents and delays. Extensive research was undertaken jointly by the steel industry and the railroads and it was found possible to eliminate the transverse fissure. Slow and controlled cooling of the rail after rolling prevents the formation of shatter cracks from which transverse fissures develop in service. Today nearly all rail is purchased to a specification with a clause requiring "controlled cooling." Additional requirements are sometimes added to rail specifications to improve the quality of rail for high-speed and heavy-traffic track and thus insure safety. Increased top discard from ingots and progressive nick and break tests to insure freedom from pipe and segregation in rail may be mentioned in this connection. These precautions insure greater uniformity of quality throughout rail melts and thereby increase dependability.

All of the many improvements in material that have been evolved by research activities have required constant revision of specifications and the writing of many new specifications. This has demanded cooperative work between producer and consumer representatives and in our Society the technical committees have devoted much time to the study and discussion of this specification work. Associated with all these researches are the names of many prominent members of this Society.

It would be inappropriate for me to discuss the increased costs the railroads of the country have had to bear for the new and higher grades of materials demanded by these specifications. However, it may be said that while "eternal vigilance is the price of safety," the price goes up rapidly with increase in speed and this refers to the price in dollars as well as in vigilance.

No discussion involving the efficiency of steam locomotives can be concluded without calling attention to the part played by water and its relation to speed and safety. Water has always played an important part as an engineering material and as an essential factor in the production of high-pressure steam it demands careful consideration.

While specifications have not been written for water used in locomotive boilers, because it is a product of nature and must be used where found, it can be modified by treatment to improve quality, thus increasing efficiency and preserving the metal of the boiler. Without such improvement, high speeds and heavy tonnage handled by the modern locomotives would be impossible. Nor would the operation of high-pressure boilers be successful.

Corrosion in locomotive boilers has been a critical problem for many years and has received much study. The results of such study have been pronounced. Even in districts having bad natural waters, corrosion is now under such complete control through scientific treatment that it has become secondary to troubles arising from fuel combustion. Freedom from boiler failures has contributed much to the safety of locomotive operation and maintenance. The result is that increased pressure and higher speeds are not only permissable but are the general rule. Although boiler pressures have been increased, boiler explosions have been notably reduced by better water conditioning.

The necessity for the steam locomotive to compete with Diesel power units with electric-motor drive has stimulated research on water for steam production. A.S.T.M. Committee D-19 on Water for Industrial Uses has been active in the study of boiler feed water and has cooperated with the Joint Research Committee on Boiler Feed Water Studies sponsored by the engineering societies and the American Association of Railroads. The personnel of these committees includes many of the ablest research men in this field.

Attention is being given to methods of testing and to the effect of water and water treatment on the metallurgical properties of boiler steels. Results of great value may be expected from this work.

Although this discussion has pertained to rail transportation, the same high standards of engineering research have perfected the automobile and made almost unbelievable strides in speed and safety of the airplane. These two forms of power are now playing a new role, and our own safety through national defense is largely dependent on these two machines. The automotive type engine has made possible the development of the modern armored tank which has wrought such overwhelming destruction in the present conflict.

In the existing national emergency and in the years ahead, the safety of this nation and the future success of our whole economic life will be determined by the work of the research scientist and the production engineer. As they have served so invaluably in the past four decades, so now and in the years to come, the specifications and the research work of the American Society for Testing

Materials will have a prominent place in solving the many grave problems that will be encountered—problems that must be successfully solved if this nation and the American way of life are to survive!

The services of this Society have been offered to our Government and we are proud that such services have already been accepted and are being used to advantage. Whatever may be the political or economic beliefs of any individual member, I can say with assurance that the A.S.T.M. membership will not be found wanting and will put forth every possible effort to speed all production in the interest of national safety.

At the present time, revolutionary changes are taking place in the world and these changes will result in a different world from that in which we have lived. Never again will we live in the world of the past two decades. The members of this Society and of similar scientific organizations must accept their responsibilities and they will play a great part in reshaping the world that is ahead of us and in solving the problems which must come.

"The moving finger writes, and having writ
Moves on; Nor all your piety nor wit
Can lure it back to cancel half a line
Nor all your tears wash out a word of it."

ANNUAL REPORT OF THE EXECUTIVE COMMITTEE

In its report to the Society at the Annual Meeting a year ago,[1] the Executive Committee presented a brief statement of the Society's potential relationship to the then newly inaugurated National Defense Program, stressing the importance of materials standards in that program and advising of various steps that had been taken to make the standardization and other facilities of the Society readily available in the event of emergency. At that meeting we listened to an address[2] by Lt-Col. Wm. C. Young on the subject of Materials Standards in National Preparedness, which was almost prophetic in its treatment of the subject in the light of the happenings of the year that has elapsed. Now, at the close of a year of defense effort, our country is under an unlimited national emergency. This unquestionably marks a turning point in the country's war effort; production for defense will be greatly intensified and will take precedence over general industrial and civilian needs; many adjustments in the usual ways of life will be required; and, considering especially our own Society activities, many ways will develop in which we can aid greatly in the intensified efforts that lie ahead. It is fitting, therefore, that the Executive Committee begin this annual report with a statement of the Society's relationship to certain phases of the defense program, a record of its contributions to the program to date, and a brief discussion of future obligations.

It has become increasingly clear that the program of production of armament and other material necessary to the national defense will demand the utmost effective utilization of our country's facilities, material, and personnel. The magnitude of the procurement problems involved are now evident. An essential element of procurement of a product or material is an adequate specification describing the desired quality, properties, and performance characteristics based upon definitive tests; and it is with this phase of the defense program as related to engineering materials that our Society is most concerned and can be of most assistance.

The nation engages upon the defense program far better prepared with respect to specifications than ever before. In the last two decades, research in industry, by the Government, and in educational institutions and research foundations has brought forth a great fund of knowledge concerning materials and methods of determining their properties and evaluating their service characteristics. This knowledge has been systematically assembled and codified into the useful form of methods of testing and specifications. By providing a forum for cooperation of industry—both producer and consumer—Government and the educational and "general interest" groups, our Society has been able to formulate "standard" A.S.T.M. methods and specifications that represent accepted industrial practice in the production and utilization of the materials covered. In 1917 there were 133 A.S.T.M. specifications, methods, and definitions; now there are 952, an increase of some seven fold, which is one measure of the progress that has been made in this work.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 57 (1940).

[2] ASTM BULLETIN, No. 105, August, 1940, p. 17.

In this same period, the Government has greatly improved the basis of its purchases by establishing the system of Federal specifications and coordinating the purchase requirements of the various Governmental departments through the activities of the Federal Specifications Executive Committee and the Procurement Division, Treasury Department. There are now over 1300 Federal specifications in addition to the hundreds of specifications of the Army and Navy covering materials primarily for military purposes. Thus, the Government is well prepared with specifications upon which to base the procurement of defense material.

Procurement of materials for defense under the stress of emergency conditions and with the utmost need for greatest speed consistent with the requisite quality puts a premium upon the soundness and practicability of specifications upon which procurement is based. It will contribute immeasurably to the ultimate success of the defense program if these specifications are critically reviewed to make sure they are abreast of modern production methods and do not contain requirements that seriously hinder procurement without commensurate benefits. To this end the Society has offered its assistance to the appropriate agencies of the Government, with the realization that as conditions with respect to availability of various strategic materials change it will be incumbent upon all specification-writing bodies to modify specifications to meet the changed conditions. Thus, the Executive Committee considers it of first importance that the Society continue vigorously the development of its standards for materials, especially those of importance in national defense; that they be kept up to date, and especially so with respect to changes such as those brought about by scarcity of certain materials; and finally, that standards be issued promptly and brought directly to the attention of those both in industry and in Government who are responsible for production and procurement of materials.

Scarcely less important than standardization itself is continued research in materials, since such research provides the knowledge required for the efficient utilization of materials and at the same time produces the factual information needed to write useful specifications. Every effort will be made to carry this work forward as expeditiously as possible, despite the fact that the essentially cooperative nature of A.S.T.M. research activities and its dependence largely upon industry for laboratory and other facilities may result in delays due to preoccupation of personnel because of production demands upon industry.

The Society's committee organization can be a real aid at this time, comprising as it does some 60 committees with a membership of over 3500 individuals and companies, representing producing and consuming interests (including Government representatives) well informed collectively on all phases of production and use of materials, with expert knowledge of testing, specification and inspection problems, accustomed to working with one another in arriving at acceptable standards of quality and performance; and finally, prepared to cooperate to the fullest possible extent with the Government and industry in the present emergency. The services of the Society and its committees have been offered to the National Advisory Defense Commission and to the Office of Production Management, as well as to departments of the Government directly concerned with specification and procurement problems.

It seems clear that Federal specification-writing activities will increase considerably as an outcome of stepped-up requirements for defense material. These activities will comprise new specifications, the revision of existing specifications, and frequently, in the interest of economy and facilitating procurement, the harmonizing of requirements of two or more specifications covering materials for substantially the same purpose. Wherever there are comparable A.S.T.M. specifications—and this is frequently the case—the knowledge and services of our committees can be used to bring about closer coordination of A.S.T.M. and Federal specification work. It is in this connection that the coordination already effected through membership of Government departments and individuals connected therewith on A.S.T.M. committees and through joint activities with various Government bureaus and laboratories will be most helpful. Even more direct cooperation is desirable, however, particularly in the early stages of specification writing. Through such cooperation it will be possible (1) greatly to facilitate inclusion of requirements in A.S.T.M. specifications to cover the needs of Government procurement, (2) to inform Federal specification committees of considerations underlying present requirements in A.S.T.M. specifications and of the status of new specification work in progress, and (3) in general to make available to them the wealth of information and data on specifications and tests in the hands of A.S.T.M. committees. Methods of coordination along general as well as specific lines are being discussed with the appropriate Government authorities, with particular reference to urgent specification work related to the defense program.

Opportunity for such coordination will develop with respect to revisions of specifications required by the need to conserve certain strategic materials. Already the Government, through the Federal Specifications Executive Committee, in collaboration with the Office of Production Management, is promulgating "emergency alternate" specifications. Under this plan, the Federal specification itself is not changed; the alternate specification, which embodies provisions for conserving scarce materials called for in the standard, is intended for use by the various agencies of the Government in all possible cases. The Society, through its committees, is prepared to advise respecting such emergency modifications, and to consider temporary revisions in its own specifications designed to accomplish the same ends. The Executive Committee is now formulating a procedure that will facilitate such emergency revisions of A.S.T.M. standards.

The American Standards Association has announced a special procedure for development and issuance of standards urgently needed in the defense program, such standards being set up for the emergency on a provisional basis subject to later review by established procedure before adoption as American Standard. The Society has already collaborated with A.S.A. in the development of a defense emergency standard entitled "Guide for Quality Control," together with a "Control Chart Method of Analyzing Data," and has expressed to A.S.A. its readiness to develop under this procedure any such standards requested in the materials field.

Too much stress can hardly be laid upon the importance of specifications in this emergency. The country is facing today a period when specifications and methods of test will be needed quickly, when changes must be made figuratively over night, and the Society must be prepared to take the initiative in such work and carry it through vigorously and with minimum of delay. It is very gratifying, therefore, that the committee work of the year reveals so much progress in the direction of standards useful in the emergency. The spring meetings contributed greatly to this progress since, being in Washington, it was possible for many representatives of the Government departments to attend and discuss requirements in specifications important to defense. However, so much pressing work of this kind lies ahead of the Society that we can in no sense be satisfied with the work so far accomplished but must continuously be on the alert to provide standards for materials as needed. A typical instance is with respect to tests and specifications for plastics, which are assuming great importance as possible substitutes for certain strategic metals; Committee D-20 on Plastics, which is doing pioneer work in this new field, has an important responsibility to provide much needed standard test methods with which to determine those properties of the different types of plastics that are urgently needed to evaluate their suitability in a variety of new uses.

As need arises for expert personnel required in the full development of the defense program, the Society can advise authoritatively in the field of materials. We have already advised with the Ordnance Department in the formation of Special Ordnance Advisory Committees on Paints, Varnishes and Related Products, and on Petroleum Products and Lubricants. Cooperation with the National Resources Planning Board in the development of the National Roster of Scientific and Specialized Personnel as it applies to "Testing of Materials and Materials Technology" was announced in the October issue of the ASTM BULLETIN; in this connection the Secretary-Treasurer serves on an Engineering Evaluation Committee to advise on the selection of personnel for specific projects.

Contact has been established with the National Defense Research Committee, to whom the services of the Society in the furtherance of research projects have been offered. The Society has kept in touch with the universities and colleges giving courses in engineering training for defense, calling their attention to the availability of the various compilations of A.S.T.M. standards that would be useful in these courses. The Society has also distributed the Index to Standards widely among procurement officers in the various branches of the Army and Navy and other purchase officers in the Government service, and has placed complete sets of Standards in the libraries of several of the key defense agencies.

In April the Executive Committee received from Mr. Donald M. Nelson, Director of Purchases, Office of Production Management, a request that the services of the Secretary-Treasurer of the Society be loaned to the OPM to aid in the urgent program of conservation of scarce materials on the part of the Federal Governmental agencies. It was explained that the demands of the defense program have already produced acute shortages in certain metals, and other materials may soon be expected to become scarce. The Executive Committee, appreciating the compliment extended in this request, gave careful consideration to it and offered the Secretary-Treasurer's services on the basis of full time until the work in question was organized (a matter of several weeks) and part time thereafter, with the understanding that his responsibilities to the Society will need to be maintained and that the importance of the activities of the Society and the Secretary's relation thereto are such that the Executive Committee reserved the privilege of reviewing the situation periodically.

Mr. Warwick began his duties with OPM on April 23 as Consultant on Specifications for Strategic Materials in the Division of Purchases. His work has to do particularly with the conservation of materials in relation to purchases by the various Governmental agencies, in which emergency revisions of the Federal and other Government specifications will be an increasingly important factor. Having previously offered its services to the OPM in furtherance of the defense program through its research and standardization activities, the Society through the Secretary's new relationship is in a position to render these services promptly and effectively. The value of A.S.T.M. specifications to industry and the Government in the present emergency is recognized and many opportunities for effective contributions by the Society to the whole problem of specifications for and procurement of defense materials will undoubtedly develop.

During the period of the Secretary's service in OPM, provision has been made for additional editorial and clerical help at Society headquarters. The Assistant Secretary, R. E. Hess, is in charge of affairs at the Society headquarters when the Secretary is in Washington.

Committee Activities:

The reports of Society committees for the year that ends with this annual meeting will comprise a record of much accomplishment in the development of new standards and revisions of existing standards and the continuance of the many research projects. In many fields of work, as mentioned above, the defense emergency has spurred our committees to unusual endeavors, to meet the needs of the moment for methods and specifications that are fully abreast of the latest technical developments. Accordingly the 1941 Supplements to the Book of Standards are expected to be larger than would otherwise be the case. Moreover many of the committee reports as printed in the *Proceedings* will contain records of important investigations carried on during the year.

The current reports of Committees E-9 on Research and E-10 on Standards deal with certain phases of the committee work and, supplemented by reviews that will appear in the August and October issues of the ASTM BULLETIN, give a general summary of the committees' activities. This report, therefore, makes brief reference only to several administrative actions during the year.

In order to simplify the work of the Society in development of specifications and tests for electrodeposited metallic coatings and also the Society's cooperative relations with the American Electro-Platers' Society and the National Bureau of Standards in work on this subject, there was organized at the spring meeting in March a new Committee B-8 on Electrodeposited Metallic Coatings under the following officers: E. M. Baker, chairman; E. A. Anderson, vice-chairman; Gustaf Soderberg, secretary. As noted in the last annual report[3] the formation of this new committee necessitated relinquishment of their jurisdiction over certain phases of the subject by the three committees who have heretofore been dealing with electroplated coatings, namely, A-5 on Corrosion of Iron and Steel, B-3 on Corrosion of Non-Ferrous Metals and Alloys, and B-6 on Die-Cast Metals and Alloys.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 43, (1940).

Jurisdiction in the matter of standards for electrodeposited coatings has been transferred from these three committees to the new committee, with the exception that Committee A-5 has retained jurisdiction for specifications for electrodeposited coatings of cadmium and zinc on steel inasmuch as it is extremely difficult to distinguish between electroplated zinc or cadmium coatings applied to steel for the purpose of finish and electroplated coatings of the same materials applied for protection purposes. Two tentative standards have been transferred from the jurisdiction of Committee A-5 to that of the new committee, namely, Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 40 T) and Tentative Methods of Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 – 40 T). The following is the formal statement of scope of the new committee:

"To prepare specifications for electrodeposited metallic coatings of all kinds on all types of base materials and methods of testing these coatings, including all pertinent investigation on the base materials, the methods of production and subsequent treatment of electrodeposits, the properties of these deposits and their performance under conditions of use and under test; excluding only electrodeposited coatings of zinc and cadmium on steel."

The Joint Committee on Exposure Tests of Plating on the Non-Ferrous Metals formerly under the auspices of the National Bureau of Standards, American Electro-Platers' Society and the A.S.T.M. has now been discharged since its activities will be carried on in the newly organized Committee B-8.

The reorganization of Committee C-18 on Natural Building Stones and Slate referred to in the report a year ago has been completed under the following officers: W. C. Clark, chairman; T. I. Coe, vice-chairman; D. W. Kessler, secretary. Since in the belief of the committee "slate" is properly understood to be included under the term "natural building stones," the title of the committee has been changed to Natural Building Stones and the statement of scope edited accordingly.

In response to a request from the Insulation Board Institute that the Society undertake the development of tests and specifications for thermal insulating board, it was decided that such work should be initiated in Committee C-16 on Thermal Insulating Materials, for which purpose the scope of that committee was enlarged to include thermal insulating materials used in the building field. The committee has already begun work on this subject and has recognized especially the interest of Committee D-6 on Paper and Paper Products in this subject by providing for formal representation of that committee on Committee C-16. The scope of Committee C-16 now reads:

"Formulation of test methods and specifications relating to thermal insulating materials, except those used in the insulating refractory field."

The scope of Committee B-6 on Die-Cast Metals and Alloys has been enlarged to include copper, iron, and other die-casting alloys. The revised statement of scope is as follows:

"Aluminum, copper, iron, lead, magnesium, tin and zinc-base die-casting alloys or any other alloys cast in molds or dies under pressures other than gravity and usually substantially greater than atmospheric pressure. Formulation of specifications and methods of test. Studies of finishing properties and corrosion resistance of die castings. Also studies of the art of die casting and the material needs for die-casting machines and dies."

The scope of Committee D-5 on Coal and Coke has been extended to include classification of coals for domestic and industrial use on the basis of chemical and physical characteristics. This has been necessitated by the fact that the Sectional Committee on Classification of Coal functioning under the procedure of the American Standards Association was discharged in February, 1939, and its work turned over to Committee D-5.

The development of standards dealing with the stabilization of soils for highway purposes is a matter of interest to both Committees D-4 on Road and Paving Materials and D-18 on Soils for Engineering Purposes. The latter committee, through its Subcommittee V on Methods of Testing for Stabilized Soils, has developed various methods of test which have important applications to the use of soils as a road material and therefore bear upon phases of the work of Committee D-4. The following policy has accordingly been set up to guide the relationship of the two committees in this subject of common interest:

> "Work on methods of testing for stabilized soils will continue under the administrative guidance of Committee D-18 through its Subcommittee V. With respect to tests of stabilized soils that have applicability as road materials, Subcommittee V will function with mutual responsibility to Committees D-18 and D-4 and will report simultaneously to the two committees. Committee D-18 will report the work of Subcommittee V to the Society and will indicate in its report the opinions and votes of Committee D-4. Committee D-4 will include in its annual report a record of its action in all such matters. The personnel of Subcommittee V shall be acceptable to the two committees. A.S.T.M. methods developed under this procedure will be indicated as being under the joint jurisdiction of Committees D-18 and D-4."

The Executive Committee approved the discharge of Subcommittee V on Magnetic Analysis of Committee A-6 on Magnetic Properties, in view of the explanation of Committee A-6 that the present state of the art of magnetic analysis does not lend itself to standardization and that as a result of the highly competitive nature of present developments, cooperative research on the part of the committee is not practicable. It was understood, however, that Committee A-6 is prepared to take up at a later time any work in the field of magnetic analysis that may be necessary.

Marking Requirements in A.S.T.M. Specifications:

During the year the Executive Committee and Committee E-10 on Standards have considered a suggestion that the clauses in A.S.T.M. specifications dealing with requirements for marking materials and products should contain a requirement that such materials or products be marked or tagged in some suitable manner to indicate conformity with the specifications. This requirement is already included in a number of instances and the practice might be advantageously extended. While this proposal necessarily has its limitations and it may not always be feasible clearly to identify material as conforming to A.S.T.M. specifications, it would seem to be of direct value and assistance to the consumer to have such requirements included in the specifications of the Society. Moreover this practice if generally followed should further enhance the prestige of A.S.T.M. specifications and promote their use. Accordingly the Executive Committee, acting jointly with the Committee on Standards, has formulated the following policy:

> "Standing committees may, wherever possible and solely for the purpose of identifying the material or product, provide in A.S.T.M. specifications that the material or product covered therein be suitably

marked or tagged with the A.S.T.M. serial designation of the specification and the type, class or grade of material or product where such is specified."

It is emphasized that the purpose of this requirement is to provide identification of the material or product as furnished by the manufacturer in accordance with the requirements of the specifications and that the Society is not in any sense certifying or guaranteeing compliance with these requirements. The latter is properly a matter entirely between the parties to the specification.

The standing committees have been requested to review the specifications in their charge and determine prior to the 1942 annual meeting whether marking requirements in accordance with this policy, if not already provided, shall be included; and if so to include the necessary revisions in their 1942 reports. In this way ample time is given for the matter to be considered and actions by the Society on such recommendations are timed to coincide with the publication of the Book of Standards.

District Committees:

The Executive Committee has noted with appreciation the activities of the district committees throughout the year which have served in a number of ways to promote the interests of the Society. In particular the holding of district meetings, as noted later in this report, has served to bring the work of the Society favorably to the attention of many individuals and companies throughout these districts and to spread a knowledge of the value and significance of the Society's standards and of the advantages of A.S.T.M. membership. Clearly the functions of our district committees are assuming added importance from year to year, providing as they do an opportunity for local activities in which members of the Society can participate as well as an excellent means of promoting the interests and welfare of the Society in the respective districts.

The fifth conference of officers of district committees was held during the spring meeting in Washington in March, 1941, at which a very informative discussion of matters of interest to the district committees took place.

While the Secretary-Treasurer or some member of the Staff meets in general at least once a year with the district committees or attends local meetings under their auspices, this has not been practicable in the case of the Northern and Southern California Committees because of the time, distance, and expense involved. However, in February, 1941, the Secretary-Treasurer visited the Pacific Coast—the first visit since September, 1938 — and met with the Northern and Southern California District Committees for full discussion regarding many phases of the Society's work in those districts. Since distance generally precludes the possibility of Pacific Coast district officers attending the annual conferences above referred to, the Secretary's visit provided the opportunity of discussing with these officers such matters as how the influence of the Society among Pacific Coast industries can be extended, knowledge of A.S.T.M. standards and data on materials promoted, how membership can be increased, and how our Pacific Coast members individually and as a group can contribute effectively to the work of the Society notwithstanding the handicap of distance. Out of these deliberations have come valuable suggestions regarding administration of district affairs, improvements in Society publications, modifications in standardization procedure and increased participation of members in the committee work of the Society.

During this visit to the Pacific Coast the Secretary-Treasurer found opportunity to call upon a number of men engaged in industries, notably aircraft manufacturing, who should be in the Society and aiding in the development of specifications and tests for materials that they either produce or use. The officers and members of the district committees aided very effectively in this work. Some general impressions from this visit are recorded in the ASTM BULLETIN for March, 1941.

Meetings:

The various meetings held during the past year have been fully reported in the ASTM BULLETIN so it is sufficient here simply to cite them as a matter of official record, with an expression of appreciation from the Executive Committee to the many members of the committees who helped to make these meetings successful.

The twelfth Spring Meeting and meetings of committees were held at Washington, March 3 to 7, 1941, and featured two symposiums, one on New Methods for Particle Size Determinations in the Subsieve Range, sponsored by Committee E-1 on Methods of Testing, the other on Color—Its Specification and Use in Evaluating the Appearance of Materials, sponsored jointly by the Inter-Society Color Council and Committee D-1 on Paint, Varnish, Lacquer and Related Products. The total registration was 1020 (the largest to date), with over 200 committee, subcommittee and section meetings. Plans for the meeting were developed by the Washington Committee on Arrangements under the chairmanship of Vice-President G. E. F. Lundell.

The following local meetings were held under the auspices of the respective district committees: in Chicago, October 10, 1940, a joint meeting of the Chicago District Committee and the Chicago Chapter of the American Society for Metals, on the subject "Aircraft Metallurgy"; in New York, October 17, 1940, on the subject of "Glass"; in Philadelphia on November 15, 1940, on the subject "Specifications—Their Use and Abuse" and on April 29, 1941, and May 28, 1941, on the subject of "Replacement Materials," the latter dealing primarily with metals; in Cleveland, January 30, 1941, on the subject "Specifications—Their Extreme Importance in the Present Emergency"; in Los Angeles, February 20, 1941, at which a lecture was presented on "Materials Testing Problems Arising from the Advance in Aircraft Performance"; in Detroit, April 18, 1941, devoted to the general subject "Changes in Materials Due to Defense Requirements," which were discussed under the headings of non-ferrous metals, chemicals and plastics, rubber, and ferrous metals. Accounts of each of these meetings have appeared or will appear in the ASTM BULLETIN.

The Executive Committee announces that the next Spring Meeting and group committee meetings will be held in Cleveland, March 2 to 6, 1942. The next Annual Meeting will be held in Atlantic City, N. J., June 22 to 26, 1942.

Publications:

While the magnitude of publications during the past year was cut down somewhat over the previous year in view of the fact that Supplements only to the Book of Standards were issued rather than complete volumes, nevertheless due to the appearance of a number of special compilations it was an exceedingly busy year. It is most significant how the demand for these special compilations continues to grow. Nine of these were issued during the past year. The sizes, editions, and number of standards in each of these publications is indicated:

A.S.T.M. Standards on Cement (Committee C-1), 107 pages, 2250 copies, 12 standards, published annually.

Manual of A.S.T.M. Standards on Refractory Materials (Committee C-8), 184 pages, 2500 copies, 25 standards, published biennially.

A.S.T.M. Standards on Paint, Varnish, Lacquer, and Related Products (Committee D-1), 356 pages, 1500 copies, 106 standards.

A.S.T.M. Standards on Petroleum Products and Lubricants (Committee D-2), 364 pages, 5000 copies, 77 standards, published annually.

A.S.T.M. Standards on Coal and Coke (Committee D-5), 136 pages, 1500 copies, 28 standards, published biennially.

A.S.T.M. Standards on Electrical Insulating Materials (Committee D-9), 344 pages, 750 copies, 47 standards, published annually.

A.S.T.M. Standards on Rubber Products (Committee D-11), 264 pages, 1200 copies, 34 standards, published biennially.

A.S.T.M. Standards on Textile Materials (Committee D-13), 380 pages, 1500 copies, 65 standards, published annually.

A.S.T.M. Specifications for Pipe and Piping Materials for High-Temperature and High-Pressure Services (Committee A-1), 153 pages, plus 2 insert plates, 1000 copies, 23 standards, published annually.

This list will be still further augmented since several other committees, notably, Committees B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, B-5 on Copper and Copper Alloys, Cast and Wrought, and C-9 on Concrete and Concrete Aggregates, have expressed an interest in having compilations issued in the fields covered by these committees.

One of the uses to which these special compilations are put is in connection with the defense courses that are now being sponsored at a number of engineering schools throughout the country. Of even more importance in this connection, however, is the special compilation of Selected A.S.T.M. Standards for Students in Engineering that is issued specifically for student use. This increased use has been reflected in an unusually heavy demand which has necessitated a further printing of the compilation this year (3000 copies), despite the fact that only two years ago an edition, that was expected to last for over three years, was struck off.

The Supplements to the Book of Standards proved to be somewhat larger than expected. This is the first experience we have had with Supplements under the new scheme of publication whereby both standards and tentative standards are included in the three parts of the Book of Standards, and in the Supplements thereto. The Supplements in 1940 contained all those standards that had been revised and adopted in that year, and all new and revised tentative standards. The new scheme has proved to be quite popular, and by having the Index to Standards and Tentative Standards appear concurrently with the Supplements, the utility of the books is greatly augmented.

The following regular publications were issued and distributed to the members:

1940 *Proceedings*, 1403 pages, 4850 copies. In addition preprints of reports and papers totaled 1209 pages, 1500 copies.

1940 Supplements to Book of Standards:
  Part I, 501 pages, 8200 copies.
  Part II, 371 pages, 6300 copies.
  Part III, 597 pages, 6100 copies.

Index to Standards, Including Tentative Standards (1940), 160 pages, 16,500 copies.

Index to Standards, Including Tentative Standards (1940-1941), 172 pages, 15,500 copies.

Year Book, 320 pages, 3350 copies.

ASTM BULLETIN, six issues, August 1940 to May 1941, 416 pages; average edition, 6700 copies.

The Symposium on New Materials in Transportation, held at the Detroit Regional Meeting in 1940 was published and furnished to members on order. The symposium comprised 100 pages, and 2000 copies were printed.

The following symposiums, presented at the 1940 annual meeting, were published in the January and March, 1941, issues of the ASTM BULLETIN, respectively:

Symposium on Tools of Analytical Chemistry
Symposium on Spectrographic Analysis

The Symposium on Spectrographic Analysis, together with the Symposium on Tools, has also been reprinted in separate pamphlet form, the size of the combined symposiums being 56 pp.; 1000 copies of the reprint were issued.

The Topical Discussion on Radiographic Testing of Airplane Components, due to lack of availability of some of the material, was not printed, but the paper and discussion at hand were mimeographed and will be furnished to the members on request.

A statement of the volume of the principal publications for the past four years follows. This statement is not truly comparative because all of the standards published in 1937 and most of those in 1938 were in the single-column format that has now been discarded:

Publication	1937, pages	1938, pages	1939, pages	1940, pages
Proceedings	2092	2084	1352	1403
Book of A.S.T.M. Standards	....	....	3794	....
Supplement to Book of A.S.T.M. Standards	282	251	....	1445
A.S.T.M. Methods of Chemical Analysis of Metals	260	35	256	....
Index to A.S.T.M. Standards and Tentative Standards	120	128	160	172
Year Book	272	286	304	320
Book of A.S.T.M. Tentative Standards	1664	1796	....	....
Spring Meeting Symposiums	232	57	251	100
	4922	4637	6117	3440

The following publications were also issued, the first two being reprints from the Proceedings:

Report of Committee A-5 on Corrosion of Iron and Steel, 25 pages, 500 copies.

Symposium on Significance of the Tension Test in Metals in Relation to Design, 113 pages, 2000 copies.

A.S.T.M. Manual on Presentation of Data (E-1), 81 pages, 2600 copies.

Index to Literature on Spectrochemical Analysis (Second Edition), 96 pages, 1500 copies.

A.S.T.M. Specifications and Methods of Test (Referred to in the Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete), 133 pages, 1500 copies.

Viscosity Index Tables, 35 pages, 2300 copies.

Viscosity Conversion Tables, 12 pages, 2300 copies.

Viscosity Temperature Chart B, 1 sheet, 20,000 copies.

A special publication covering a Report on Significance of Tests of Concrete and Concrete Aggregates, issued in 1935, is now being brought up to date under the sponsorship of a committee headed by Mr. R. W. Crum. Several chapters of this publication are being presented at the current annual meeting as individual papers.

A further publication is now under revision, namely, the Symposium on Mineral Aggregates issued in 1929. Mr. Stanton Walker is chairman of a special committee in charge of this revision.

Work is now in progress on the Index to Technical Reports and Papers (1936–1940). This is an extension of the former five-year Index to Proceedings, and will include references to papers published in the ASTM BULLETIN and any special publications, as well as those appearing in the *Proceedings*. It is expected that this Index will be available later in the year.

Charge for Right to Reprint Standards.—Mention was made in the 1940 report of the Executive Committee of the establishment of a policy of charging for the reprinting of A.S.T.M. Standards. In this connection a number of requests have been received during the year for permission to reprint. It is, of course, understood that this applies only to the reprinting of standards where they are used for commercial distribution; no charge is made for the reprinting of standards in text books and similar publications.

Registration of A.S.T.M. Seal:

The A.S.T.M. seal has been registered as a trade mark by the U. S. Patent Office under certificate of registration dated October 15, 1940. It is the intention to reserve the use of the Society seal solely for publications issued by the Society. Wherever the seal is used in the title pages of publications and in the ASTM BULLETIN the fact of registration will hereafter be indicated.

Membership:

The membership of the Society on June 1, 1941, was 4470, which is the highest in our history—the previous high having been 4449 in October, 1930. Statistics showing gains and losses in various classes of membership are as follows:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1940	June 1, 1941	Resignations	Dropped	Deaths	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	6	9	...	...	...	...	3	...	...	3	3
Life or Perpetuity	10	9	...	...	1	...	...	...	1	...	1[a]
Sustaining	88	136	...	...	...	...	39	9	...	48	48
Company, Firm, etc.	1007	1013	16	7	...	45	6	68	68	74	6
Individual, etc.	3140	3225	113	47	22	9	19	257	191	276	85
Junior	63	78	4	4	...	13	...	36	21	36	15
Total	4314	4470	133	58	23	67	67	370	281	437	156
Student	411	437	42	187	...	19	...	274	248	274	26

[a] Loss.

The net gain for the year of 156 compares with 115 for the preceding year and 43 for two years ago. Elections to membership were 370 compared to 358 for the preceding year; losses from resignations and delinquencies were 191 compared to 222 for the preceding year. The number of student members increased from 411 to 437.

The growth of sustaining membership has continued in a very gratifying manner, the number of sustaining members having increased from 88 a year ago to 136 as of June 1. The names of the 48 new sustaining members, listed alphabetically are given below:

Acme Steel Co.
American Brass Co., The
American Cast Iron Pipe Co.
American Cyanamid Co.
American Gas and Electric Service Corp.
American Rolling Mill Co., The
Armstrong Cork Co.
Babcock & Wilcox Co., The
Barrett Co., The
Bigelow-Sanford Carpet Co., Inc.
Canadian Car and Foundry Co., Ltd.
Chapman Valve Manufacturing Co., The
Chrysler Corp.
Combustion Engineering Co., Inc.
Consolidated Gas Electric Light and Power Co. of Baltimore
Dow Chemical Co., The
Flintkote Co., The
General Electric X-Ray Corp.
Hercules Powder Co.
Hygrade Sylvania Corp.
Irvington Varnish and Insulator Co.
Jones & Laughlin Steel Corp.
Kimble Glass Co.
Laclede Steel Co.
Lebanon Steel Foundry
Monsanto Chemical Co.
Montgomery Ward and Co.
National Cash Register Co., The
National Gypsum Co.
Pennsylvania Railroad Co., The
Revere Copper and Brass, Incorporated
Riverside Metal Co., The
Rustless Iron and Steel Corp.

Scovill Manufacturing Co.
Smith and Sons Carpet Co., Alexander
Southwestern Portland Cement Co.
Sperry Gyroscope Co., Inc.
Superheater Co., The
Tennessee Coal, Iron and Railroad Co.
Tennessee Eastman Corp.
Timken Roller Bearing Co., Steel and Tube Division
Udylite Corp., The
Union Pacific Railroad Co.
United Engineering and Foundry Co.
United States Pipe and Foundry Co.
Westinghouse Air Brake Co.
Westinghouse Electric and Manufacturing Co.
York Ice Machinery Corp.

The Riverton Lime and Stone Co., Inc., has qualified as a sustaining member since June 1 and Chase Brass & Copper Co. will become a sustaining member shortly.

Of these, as may be seen from the accompanying membership table, 39 were acquired by transfer of existing company memberships (which accounts for the relatively small net increase in the number of company memberships during the year) and nine were acquired by companies who had not previously held company membership. The Executive Committee expresses on behalf of the Society its thanks to these companies for their willingness to lend increased financial support to the Society's activities through this form of membership.

The Executive Committee also expresses its thanks to the district committees and the many members of the Society who throughout the year have aided so effectively in the acquiring of new members.

The Executive Committee has authorized the Secretary-Treasurer to waive upon their request the dues of members of the Society who have entered the armed forces of their country. The names of such members will be retained on the membership rolls during the period of their service and they will retain all privileges of membership, save that the Book of Standards and the *Proceedings* will not be sent them. They will continue to receive the ASTM BULLETIN. Regarding membership on standing committees of members in service, the standing committees are privileged to take cognizance of any unusual conditions of this kind affecting the activity of their committee members and, if they desire, to place such members in an inactive status for the duration of the emergency.

The Society has lost the following members through death:

	Date of Membership
Crawford, W. W.	1926
Dickason, H. T.	1928
Hadfield, Robert A.	1906
Heckel, G. B.	1905
Hovey, Otis E.	1908
Hutchins, R. T.	1939
Johnson, A. N.	1903
Jumper, Charles H.	1922
Lasier, Emery L.	1912
Law, LeRoy M.	1912
McKinney, Paul E.	1915
Muckenfuss, A. Moultrie	1909
Payne, M.	1921
Reed-Lewis, E. W.	1935
Sabin, Alvah Horton	1898
Schorndorfer, S. C.	1928
Stolzenbach, William F.	1937
Storey, W. B.	1903
Thomas, David	1900
Uhlig, Edward C.	1925
von Frankenberg und Ludwissdorf, Albrecht	1934
Wirtz, Max F.	1931
Young, W. W.	1908

Many of these members were engaged in the technical and administrative work of the Society, among them G. B. Heckel, a former member of the Executive Committee, A. N. Johnson, a former member of the Committee on Papers and Publications, E. L. Lasier, L. M. Law, P. E. McKinney, A. M. Muckenfuss, and A. H. Sabin.

At the annual meeting the Executive Committee will award a certificate commemorating forty years of continuous service to C. N. Forrest. Twenty-seven such awards have been made since these awards were established three years ago.

Finances:

Report for the Fiscal Year 1940.—The annual statement of the finances of the Society follows in the report of the auditors for the fiscal year, January 1 to December 31, 1940. The report gives the balance sheet of assets and liabilities, including special and designated funds (that is, the Research Fund, Medal and Lecture Fund, and Committee Funds); statement of receipts and disbursements, in which items are classified into "operating" (budgeted) and "nonoperating"; details of special funds; and investment of Society funds, both book and market values being given.

Total receipts from dues and entrance fees in 1940 were $91,928.53, an increase of nearly $9000 over the preceding year—a fact that is accounted for by a substantial growth of membership and the very substantial increase in the number of sustaining members. Receipts from sales of publications were at the all-time high total of $81,638.65, which is due primarily to the heavy sales of the 1939 edition of the Book of Standards, which amounted to practically $36,500. This, together with the largest sale of separate reprints of standards in any year, approximately $9200, is convincing evidence of the important place that A.S.T.M. standards fill in the industrial world. Miscellaneous receipts amounted to $15,311.96, consisting principally of receipts from advertising in the ASTM BULLETIN and Index to Standards and Tentative Standards, interest and dividends, and registration fees. Receipts from advertising in the BULLETIN, nearly $7200, slightly exceeded those in 1937, the highest previous year.

Total operating receipts were $188,879.14. Total operating disbursements with all current bills paid (as detailed in the auditors' report) were $164,623.39. This sum includes $6000 applied from current income towards the reserve for publication of the 1942 Book of Standards; also $1500 (one-half of the entrance fees) paid into the principal of the Research Fund and $1259.30 paid into the Employees Retirement Plan.

A favorable balance between operating receipts and disbursements of $24,255.75 has resulted. It is to be remembered that the Society's financial operations need to be considered over a three-year period for a true picture because of the factors introduced by triennial publication of the Book of Standards. In view of the uncertain times that lie ahead it is gratifying that the three-year period 1940–41–42 has gotten off to such an excellent start through this favorable balance. It is likely that, in line with experience in preceding triennial fiscal periods, some of this surplus will be needed for operation of the Society in 1941 and 1942.

Attention is called to the payment, as noted under nonbudgetary disbursements, of $10,109.75 from Book of Standards Reserve. This completes the payment of costs on the 1939 Book of Standards, which as reported a year ago totaled $27,088.92. There remains a balance in the Book of Standards Reserve of $1890.25 which will be applied to costs of the 1942 edition.

Attention is also directed to the cost of publishing the 1940 Supplements to the Book of Standards comprising 501 pages, Part I (8200 copies); 371 pages, Part II (6300 copies); 597 pages, Part III (6100 copies), namely $14,928.13.

The auditors' report includes a statement of the financial condition of the Society for the past five years. The surplus in the general funds account, exclusive of special and designated funds, is $81,144.08, which compares with $58,117.14 a year ago. The principal of the Research Fund as of December 31, 1940, was $28,791.15.

Investment of Society funds as of December 31, 1940, are given in the auditors' report, both book and market values being shown. The following changes in the investment portfolio, including the purchase of a number of dividend-paying common stocks from surplus, are noted:

GENERAL FUNDS

Sold		Received
$3000	Penna. State Water Corp. First Collateral trust, 1966 (called)	$3 192.62
20	shares West Penn Power Co. Preferred	2 338.60
1000	Columbia Gas & Electric Co. Debenture 5's, due 1961 (sold to Research Fund)	1 012.66
3000	Republic Steel Corp. General Mortgage B, 4½'s, due 1961 (Transferred to Research Fund)	....
1	share Standard Oil Co. of New Jersey	40.68
		$6 584.56

Bought		Cost
30	shares Alvin Progressive Federal Savings & Loan Assn.	$3 000.00
30	shares Collingdale Federal Savings & Loan Assn.	3 000.00
50	shares American Cyanamid Co., Class B, common	1 764.25
7	shares American Cyanamid Co., 5 per cent Cumulative Convertible Preferred (dividend)	....
100	shares Atlantic Refining Co., common	2 302.50
100	shares Borden Co.	1 989.00
25	shares Colgate-Palmolive-Peet Co. $4.25 Preferred	2 525.00
50	shares General Telephone Corp. $2.50 Preferred	2 675.00
100	shares Mathieson Alkali Works, common	3 016.00
25	shares J. C. Penney Co., common	2 279.63
100	shares Public Service Corp. of N. J., common	4 129.50
		$26 680.88

RESEARCH FUND

Sold		Received
$1000	Baldwin Locomotive Works First Mortgage Sinking Fund Bonds (matured)	$1 000.00

Bought		Cost
100	shares United Gas Improvement Co. common	$1 489.00
$3000	Republic Steel Corp. General Mortgage Series B, due 1961, 4½'s (Transferred from Society Funds)	....
$1000	Columbia Gas & Electric Co., Debenture 5's, due 1951	1 012.66
		$2 501.66

Profits totaling $509 were realized on sales of securities during the fiscal year.

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock. An inventory of technical publications as of June 1, 1941, may be summarized as follows:

	Number of Copies
Year Book	111
Proceedings	4 800
Index to Proceedings, 5 parts, total	1 379
1939 Book of A.S.T.M. Standards:	
Part I	1 788
Part II	1 178
Part III	1 014
Supplements to 1939 Book of A.S.T.M. Standards	
Part I	2 696
Part II	1 844
Part III	1 839
Index to Standards and Tentative Standards	986
Selected Standards for Students in Engineering	2 550
Reprints of Standards and Tentative Standards (approximate)	57 000
Special Reprints (approximate)	33 000

1941 Finances.—The Society's operations for 1941 are based upon budgeted operating receipts of $176,600, of which $172,000 is estimated current receipts and $4600 is applied to 1941 operations from surplus. Receipts from dues and entrance fees are estimated at $93,000; receipts from sales of publications at $61,500, of which about $23,000 is expected to be realized from sales of the Book of Standards and its Supplements; miscellaneous receipts, including advertising, interest and dividends and A.S.T.M. Exhibit, are estimated at $17,500. Disbursements have been estimated at $175,650, leaving $950 for contingencies. The budget provides fully for all the Society's normal operating expenses, including all current publications, activities of standing and district committees, as well as additions to the clerical staff required by the growth of Society activities. It also includes the sum of $1000 to be applied to expenses that might need to be incurred in relation to the emergency program on national defense.

The Executive Committee reviews the budget of receipts and disbursements quarterly and makes any adjustments that may be necessary.

The following investments have been

made so far this year: Twenty-five shares of common stock of American Can Corp. costing $2122.30 were bought for the General Account and 25 shares of common stock of Pittsburgh Plate Glass Co. costing $1955 were bought for the Research Fund. The bonds of the Wheeling Steel Co. of which the Society had $3000 in the General Funds have been called, and the proceeds, augmented by funds from current income, have been invested in $4000 General Steel Castings Corp. First Mortgage, Series A, 5½ per cent Bonds, and $3000 Pressed Steel Car 15-yr. 5 per cent Debenture Bonds. These investments do not appear in the current auditors' report. They bring the total book value of Society investments as of May 31, 1941, in all funds to $120,105.99; the market value of these securities on the same date was $94,831.29. The Society's cash balance in the General Funds on May 31, 1941, was $90,261.86.

Retirement Plan for Employees:

The Retirement Plan for A.S.T.M. Employees continues along lines that have been set forth in earlier reports. May 3, 1941, marked the close of the third year of the Plan. Beginning with that date the Executive Committee increased the Society's contribution by a total of $20 per month, comprising the first contract for a new employee and additional contracts for seven employees, most of them of long years of service. Three of the newer employees are eligible for first contracts later this year. The Society's annual contribution to the plan is now at the rate of approximately $1475, which is slightly over 2½ per cent of the salary roll. Participating employees match the Society's contribution and have the opportunity of increasing their own contributions to provide insurance features or increase the face value of the retirement income policies.

Staff Organization:

As announced in the ASTM BULLETIN for March, 1941, the Executive Committee has established the following positions and titles, and the members of the present staff whose names appear below have been appointed respectively to the positions stated:

Standards Editor	P. J. Smith
Assistant to the Secretary	R. J. Painter
Assistant Editor	G. A. Wilson

The Executive Committee has also designated the Assistant Secretary, R. E. Hess, as Editor of the BULLETIN and Mr. Painter as Associate Editor.

Proposed Amendment of the By-laws:

Experience with the method of selecting members of the Nominating Committee, as set forth in Article III of the By-laws, has led the Executive Committee to conclude that it is advisable to appoint the Nominating Committee at the October quarterly meeting of the Executive Committee instead of the January meeting, in order that there may be opportunity to announce the names of the members and alternates on the Nominating Committee well in advance of their scheduled meeting in March of each year. The Executive Committee accordingly recommends that the following amendment of Article III, Section 2, of the By-laws be referred by the annual meeting to letter ballot of the Society for adoption:

Substitute the italicized words in this section for those in brackets:

> "Section 2. *Not later than September 1* [Within five days after December 1] of each year the Secretary-Treasurer shall notify every member of the Society that he has the right to recommend two members for appointment on the nominating committee. The procedure governing the transmission of such recommendations shall be designed

REPORT OF THE AUDITORS FOR THE FISCAL YEAR, JANUARY 1, TO DECEMBER 1, 1940

Philadelphia, January 9, 1941.

MR. C. L. WARWICK, *Secretary-Treasurer*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of your Society for the year ended December 31, 1940. We did not make a detailed audit of all transactions, but made extensive test checks to determine the accuracy of your records.

We have prepared and submit herewith Balance Sheet as of December 31, 1940, Statement of Cash Receipts and Disbursements for the year ended that date and other supporting schedules, all of which are in agreement with the records of your Society. In our opinion, these statements present fairly the financial position of the Society at December 31, 1940, and the results of its operations for the year then ended.

The investments owned by the Society as detailed were examined by us. All income from investments was properly accounted for.

Respectfully submitted,

(Signed) JOHN HEINS & CO.
Certified Public Accountants

BALANCE SHEET AS OF DECEMBER 31, 1940

(Including Special and Designated Funds)

ASSETS

General Funds:			
Current: Cash	$32 635.12		
Less checks drawn as of December 31, 1940—Cost of Proceedings ($11 760) and 1940 Supplement to Book of Standards ($11 266.51) in hands of Secretary-Treasurer that date	23 026.51		
		$9 608.61	
Investments (Market Value, $62 141.24)—Cost		72 568.95	
Accounts Receivable		13 388.88	
Total Current Assets—General Fund			$95 566.44
Furniture and Fixtures			6 561.93
Total General Funds			$102 128.37
Special and Designated Funds:			
Cash: A.S.T.M. Research Fund		$2 484.65	
Dudley Medal and Marburg Lecture Fund		311.38	
Committee Funds		10 057.85	
			12 853.88
Investments:			
A.S.T.M. Research Fund (Market Value, $22 591.25)—Cost or book value		$27 400.99	
Dudley Medal and Marburg Lecture Fund (Market Value, $2430.00)—Cost		6 625.00	
Committee Funds (Market Value, $6646.25)—Cost		6 000.00	
			40 025.99
Total Special and Designated Funds			$52 879.87
Total Assets			$155 008.24

LIABILITIES

General Funds:			
Current: Accounts Payable:			
Committee A-1	$2.54		
Committee C-1	109.79		
Advance Collections	3 267.92		
Total Current Liabilities—General Fund		$3 380.25	
Life Membership Fund	$3 005.20		
Headquarters Fund	2 564.48		
Publication Fund	3 015.00		
Book of Standards Reserve	7 890.25		
Executive Retirement Reserve	1 050.00		
Retirement Fund Reserve	79.11		
Surplus	81 144.08		
		98 748.12	
Total General Funds			$102 128.37
Special and Designated Funds:			
A.S.T.M. Research Fund		$29 885.64	
Dudley Medal and Marburg Lecture Fund		6 936.38	
Committee Funds		16 057.85	
Total Special and Designated Funds			52 879.87
Total Liabilities and Funds			$155 008.24

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1936-1940, INCLUSIVE

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable and Miscellaneous	Furniture and Fixtures	Accounts Payable and Miscellaneous	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1936	$6 943.82	$29 767.23	$16 088.91	$6 915.95	$2 624.62	....	$7 061.76	$50 029.53
1937	20 686.08	37 599.49	10 506.78	7 029.87	2 140.86	$6 000.00	9 061.76	58 619.60
1938	24 425.51	39 568.24	9 609.00	6 912.96	2 283.88	12 000.00	8 898.99	57 332.84
1939	9 329.65	54 902.10	14 880.53	6 467.27	5 777.01	12 000.00	9 685.40	58 117.14
1940	9 608.61	72 568.95	13 388.88	6 561.93	3 380.25	7 890.25	9 713.79	81 144.08

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD ENDED DECEMBER 31, 1940

Cash balance, January 1, 1940			$9 329.65

RECEIPTS

Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$85 951.78		
Past Dues	843.25		
Advance Dues	1 903.50		
Income, Life Membership Fund	150.00		
Entrance Fees	3 080.00		
Total Dues		$91 928.53	
Sale of Publications:			
Separate Standards	9 218.97		
Book of Standards (Members, addl. parts)	5 698.03		
1939 Book of Standards	36 492.56		
1940 Supplements to Book of Standards	1 686.10		
Methods of Chemical Analysis of Metals	1 212.18		
Proceedings	3 346.23		
Selected Standards for Students in Engineering	1 346.10		
Bulletin Subscriptions (Non-Members)	332.35		
Special Reprints and Miscellaneous	22 306.13		
Total Sale of Publications		$81 638.65	
Miscellaneous:			
Binding (Members)	189.00		
Advertising, Bulletin	7 194.05		
Advertising, Index	1 707.50		
Interest and Dividends	3 518.65		
Registration and Other Fees, Annual Meeting	1 679.78		
Committee A-1 for Technical Assistant	945.91		
Miscellaneous	77.07		
Total Miscellaneous Items		$15 311.96	
Total Operating Receipts (Budgeted)		$188 879.14	
Nonoperating Receipts (Not Budgeted):			
Investments, Matured or Sold	$6 584.56		
Excess Remittances	282.04		
Accrued Interest	17.36		
Creep Data Volume	672.76		
Symposium on Effect of Temperature	152.41		
Committee C-1 for Technical Assistant	1 400.00		
Executive Retirement Reserve	50.00		
Retirement Fund Reserve	79.11		
Total Nonoperating Receipts		$9 238.24	
Total Receipts			$198 117.38
Carried Forward			$207 447.03

Brought forward			$207 447.03

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$2 081.75		
1940 Supplements to Book of Standards	14 928.13[a]		
Methods of Chemical Analysis of Metals	8.57		
Proceedings	15 340.71[b]		
Preprints	2 627.31		
Regional Meeting Papers	989.10		
ASTM Bulletin	7 629.23		
Circulars to Members	1 312.48		
Separate Standards	4 399.56		
Index to Standards and Tentative Standards	2 838.30		
Reprints (Reports, Papers, Symposiums, etc.)	9 195.96		
Miscellaneous Printing (Authors' Reprints, etc.)	611.46		
Resetting Standards (Double Column)	4 002.39		
Total Disbursements, Publications		$65 964.95	
Salaries		59 101.38	
General Office Expenses		12 834.63	
Expenses, Standing and District Committees		3 878.11	
Expenses, Meetings and A.S.T.M. Exhibit		3 495.86	
Traveling Expenses, Administrative and Special Committees		2 026.27	
American Standards Assn. and Sectional Committees		1 000.00	
Rent, Headquarters		6 207.06	
Furniture and Fixtures		1 032.08	
Principal, A.S.T.M. Research Fund (one-half current year's entrance fees)		1 500.00	
Employees Retirement Fund		1 259.30	
Miscellaneous (Dues, Contributions, etc.)		323.75	
Total Operating Disbursements (Budgeted)		$158 623.39	
Nonoperating Disbursements (Not Budgeted):			
Investments	$26 680.88		
Accrued Interest	17.36		
Refund of Excess Remittances	284.04		
Purchase of Retirement Income	250.00		
Book of Standards (Reserve)	10 109.75		
Creep Data Volume	323.00		
Technical Assistant, Committee C-1	1 550.00		
Total Nonoperating Disbursements		39 215.03	
Total Disbursements			$197 838.42
Cash Balance, December 31, 1940			$9 608.61

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[a] This amount includes $11,266.51 covering checks drawn against cost of 1940 Supplement to Book of Standards but not actually paid on December 31, 1940.

[b] This amount includes $11,760.00 covering checks drawn against cost of *Proceedings*, but not actually paid on December 31, 1940.

In addition to the general funds of the Society, the Secretary-Treasurer has on hand the following funds as of December 31, 1940:

A.S.T.M. RESEARCH FUND

Principal Account:	*Total*	*Invested*	*Uninvested Cash*
Balance January 1, 1940	$24 282.68	$22 890.86	$1 391.82
Receipts—Securities matured		−991.53	+1 000.00
Profit on Securities matured	+8.47		
Contribution — A.S.T.M. Current Entrance Fees	+1 500.00		+1 500.00
Contribution (securities) — A.S.T.M. Past Entrance Fees	+3 000.00	+3 000.00	
	$28 791.15	$24 899.33	$3 891.82
Disbursements—Purchase of securities		+2 501.66	−2501.66
Balance, December 31, 1940	$28 791.15	$27 400.99	$1 390.16

Income Account:		
Balance January 1, 1940	$750.73	
Receipts—Interest and Dividends	993.76	$1 744.49
Disbursements:		
Experiments on Determination of Water Vapor in Gaseous Fuels (Committee D-3)	200.00	
Study of Weldability of Carbon Steel (Engineering Foundation)	200.00	
Research Committee, Fatigue of Metals	250.00	650.00
Cash Balance, December 31, 1940		$1 094.49

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1940:			
Principal—Investments (at Cost)	$6 625.00		
Income—Cash	518.63	$7 143.63	
Receipts			
Interest and Dividends		66.00	7 209.63
Disbursements			
Engrossing 1940 Lecture Certificates		3.25	
Honorarium, 1940 Lecturer		200.00	
Cost of 1940 Medal		70.00	273.25
Balance, December 31, 1940:			
Principal—Investments (at Cost)		6 625,00	
Income—Cash		311.38	$6 936.38

COMMITTEE FUNDS

Committee A-1 on Steel	$ 729.65	
Committee A-5 on Corrosion of Iron and Steel	2 902.35	
Committee A-5 Subcommittee X on Embrittlement Investigation	141.14	
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	3 137.04	
Committee B-6 on Die-Cast Metals and Alloys	138.42	
Committee C-1 on Cement	971.23	
Committee C-1, Studies of Additions to Cement	22.95	
Committee C-1, Cement Reference Laboratory	1 012.74	
Committee C-9 on Concrete and Concrete Aggregates	189.52	
Committee C-9, Sanford E. Thompson Medal Fund	498.75	
Committee C-15 on Manufactured Masonry Units	319.36	
Committee C-18 on Natural Building Stones	174.56	
Committee D-1 on Paint, Varnish, Lacquer and Related Products	111.84	
Committee D-2 on Petroleum Products and Lubricants	87.27	
Committee D-5 on Coal and Coke	1 211.70	
Committee D-13 on Textile Materials	382.60	
Committee D-13, Testing Textile Finishes	3 584.53	
A.S.A. Sectional Committee on Specifications for Cast-Iron Pipe and Special Castings	180.12	
Research Committee on Fatigue of Metals	262.08	
Total Committee Funds		$16 057.85
Accounted for as Follows:		
Cash Balance, December 31, 1940	$10 057.85	
Investments—at Cost	6 000.00	$16 057.85

INVESTMENTS, DECEMBER 31, 1940

GENERAL FUNDS

Number of Shares or Par Value		*Book Value*	*Market Value*[a]
	Bonds		
$3000	Armour & Co. of Del., First Mortgage, 20-yr. Sinking Fund, Series C, 4's, due 1957	$2 955.00	$3 183.75
9000	Balto. & Ohio R. R., Refunding & General Mortgage 5's, Series F, due 1996	9 106.55	3 330.00
2000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	2 025.34	2 110.00
500	Florida Power & Light Co., First Mortgage, 5's, due 1954	491.25	531.25
3000	Michigan Consolidated Gas Co., First Mortgage, 4's, due 1963.	2 925.00	3 187.50
2000	North Station Industrial Bldg. Inc., First Mortgage Sinking Fund, 5's, due 1962*	1 850.00	1 540.00
4000	N. Y., Chicago & St. Louis R. R., Refunding Mortgage, Series C, 4½'s, due 1978	2 805.61	2 375.00
3000	Wheeling Steel Corp., First Mortgage Sinking Fund, Series A, 4½'s, due 1966	3 045.00	3 195.00
		$25 203.75	$19 452.50
	Stock		
7	shares American Cyanamid Co., 5 per cent Cumulative Convertible Preferred	$70.00	$82.50
50	shares American Cyanamid Co., Class B common	1 764.25	1 887.50
100	shares American Radiator & Std. Sanitary Corp., common	1 564.32	675.00
25	shares American Tobacco Co., common	2 110.12	1 743.75
100	shares Atlantic Refining Co., common, par value $25	2 302.50	2 375.00
100	shares Borden Co.	1 989.00	1 887.50
30	shares Alvin Progressive Federal Savings & Loan Assn.[b]	3 000.00	3 000.00
30	shares Collingdale Federal Savings & Loan Assn.[b]	3 000.00	3 000.00
25	shares Colgate-Palmolive-Peet Co., $4.25 Preferred	2 525.00	2 556.25
50	shares Gen. Elec. Co. common, no par value	2 006.25	1 656.23
50	shares General Telephone Corp., $2.50 Preferred*	2 675.00	2 650.00
50	shares Great Northern Ry., $6 Preferred, par $100	1 774.83	1 300.00
25	shares Hazel Atlas Glass Co.	2 510.64	2 296.88
100	shares S. S. Kresge Co., common	2 490.18	2 512.50
100	shares Mathieson Alkali Works, common, no par value	3 016.00	2 750.00
25	shares J. C. Penney Co., common	2 279.63	2 150.00
100	shares Public Service Corp. of N. J., common	4 129.50	2 937.50
25	shares Sears, Roebuck & Co., capital	1 760.71	1 953.13
200	shares United Gas Improvement Co., common	2 578.00	1 975.00
50	shares U. S. Gypsum	3 819.27	3 300.00
		$47 365.20	$42 688.74
		$72 568.95	$62 141.24

A.S.T.M. RESEARCH FUND

	Bonds		
$2000	First Mortgage Real Estate, 1315-1317 Spruce St., 6's, due 1945*	$2 000.00	$600.00
1000	Allis Chalmers Mfg. Co., Convertible Debentures, 4's, due 1952	1 035.28	1 065.00
1500	Balto. & Ohio R. R., General & Refunding, 5's, Series F, due 1996	1 511.25	555.00
2000	Columbia Gas & Electric Co., Debentures, 5's, due 1961	2 025.31	2 110.00
1500	Florida Power & Light Co., First Mortgage, 5's, due 1954	1 473.75	1 593.75
3000	North Station Industrial Bldg. Inc., First Mortgage Sinking Fund, 5's, due 1962*	2 812.50	2 310.00
2000	N. Y., Chicago & St. Louis, Refunding & Mortgage, Series C, 4½'s, due 1978	1 400.26	1 187.50
3000	Republic Steel Corp., General Mortgage, Series B, 4½'s, due 1961	3 000.00	3 135.00
		$15 258.35	$12 556.25

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[a] Market values were taken from *The Philadelphia Inquirer* with the exception of certain quotations on inactive or unlisted securities indicated by an asterisk (*). These values were obtained from brokers as of December 31, 1940.

[b] In the absence of a quoted market value these securities are included at their cost which is equal to their face value.

Stock		
12 shares Consolidated Edison Co. of N. Y., $5 Preferred	1 234.39	1 284.00
29 shares Consumers Power Co., $4.50 Preferred, no par value	2 914.50	3 059.50
98 shares General Motors Corp., common, par $10[c]	6 504.75	4 704.00
100 shares United Gas Improvement Co., common	1 489.00	987.50
	$12 142.64	$10 035.00
	$27 400.99	$22 591.25
DUDLEY MEDAL AND MARBURG LECTURE FUND		
$500 Balto. & Ohio R. R., Refunding & General Mortgage, Series C, 6's, due 1995	550.00	210.00
6000 Balto. & Ohio R. R., Refunding & General Mortgage, Series A, 5's, due 1995	6 075.00	2 220.00
	$6 625.00	$2 430.00
COMMITTEE FUNDS		
$5000 U. S. Treasury Bonds, $2\frac{3}{4}$'s, due 1947–1945	5 000.00	5 531.25
1000 U. S. Treasury Bonds, $2\frac{7}{8}$'s, due 1960–1955	1 000.00	1 115.00
	$6 000.00	$6 646.25
	$112 594.94	$93 808.74

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[c] The income from these shares is by agreement paid to an annuitant.

to ensure secrecy as to individual authorship. A committee of tellers appointed by the President shall canvass the recommendations which have been received by the Secretary-Treasurer prior to *October* [January] 1, and shall transmit the returns to the Secretary-Treasurer in time for presentation to the Executive Committee at its *October* [January] quarterly meeting . . . ." (No change in remainder of section.)

Engineering Relations Between the American Republics:

The nations of the Americas are being drawn more closely together by the force of world events. In our own country, through the State Department and the recently organized Office for Coordination of Commercial and Cultural Relations between the American Republics (the latter established as a defense agency), much is being done as recorded in the press to promote better mutual understanding of the business, industries, and cultures of the American nations and the importance of these things to the broad problems of economic integration and defense of the Western Hemisphere. It has appeared to the Executive Committee that an important part of this movement should be the cultivation of closer relationship in scientific and technical matters among the American republics. In our own field of engineering materials, such relationships are limited to approximately 78 memberships in A.S.T.M. on the part of governmental departments, companies and a few individuals from the Latin-American countries, to the exchange of publications with several of the scientific bodies of those countries, and to the exchange of standards and other data with the Argentine Institute of Standardization of Materials and, recently, with the similar body being organized in Brazil. Neither the International Association for Testing Materials nor the International Standards Association provides a satisfactory channel, even in normal times, for collaboration with the Latin-American countries in matters dealing with specifications and tests for materials. The Society has accordingly recommended to the State Department, the O.C.C.C.R. and the Pan-American Union, that consideration be given to means of promoting interchange of information on the properties of materials, progress in the development of tests and specifications, and the significance of standards for materials in the whole economy of the American republics. It has been suggested that this subject be given a place on the program of the next Pan-American Scientific Congress tentatively planned for Havana in 1945, and assurance has been given that the Society is prepared to participate in such a program and to sponsor suitable papers and reports.

In this connection, the Secretary-Treasurer presented a paper on "Inter-American Relations in the Field of Engineering Materials" at the annual meeting last December of the Engineering Section of the American Association for the Advancement of Science, which was devoted to a general discussion of the economic and engineering interests of the Americas.

In any consideration of Pan-American relationships, the barrier of differences in language needs to be faced Hence the question of translating important engigineering data into Spanish and Portuguese is of special significance. With respect to materials standards, in the early 1920's some sixty A.S.T.M. standard specifications were translated into French, Spanish, and Portuguese and published by the Department of Commerce as part of its Industrial Standards Series in the interest of promotion of our export trade. Interest in such translations is now reviving; the American Refractories Institute has translated and published, with the Society's collaboration, 3000 copies of a Spanish edition of 13 A.S.T.M. standards for refractories, and there is evidence of interest on the part of the steel industry in similar translations of a large group of A.S.T.M. specifications for steel and steel products. Further evidence of the need for such translations is seen in the request of the Portland Cement Institute of Argentina for permission to publish a Spanish edition of the Report of the Joint Committee on Concrete and Reinforced Concrete, which has of course been granted, and the publication in Spanish of A.S.T.M. standards relating to petroleum products by the Argentine Government Oil Fields.

It will be the policy of the Society to encourage translations of A.S.T.M. standards, as well as reports and papers where desired, and to collaborate with any groups interested in the publication and distribution of foreign language editions of such material. The technical and economic significance of such work, especially at the present time, is self-evident.

Cooperative Activities:

The Society's activities have always of necessity involved cooperative relations with many other organizations, which relations proceed along well-established lines. News items in the ASTM BULLETIN have told of various activities in this direction and these need not be repeated. Several happenings during the year, however, merit specific mention here.

The Society's contacts with the American Standards Association constitute one of its most important cooperative activities. The submission of new and revised standards to A.S.A. for approval as American Standard has continued during the year and reference should be made to the annual report of Committee E-10 on Standards for a detailed statement. A subject that has been under consideration is that of a recommended practice for public approval and certification procedures. The Executive Committee has commented upon a draft of a proposed recommended practice; and recognizing that there is a growing demand for certification for ultimate consumer goods the Executive Committee has appointed a small subcommittee to study this question of certification and the problems involved therein as an aid in the formulation of Society policy. As somewhat related to this subject it is significant that the Society was invited by the American Standards Association to be represented on the Advisory Committee on Ultimate Consumer Goods, which functions as the general correlating agency in this field under the auspices of A.S.A. Vice-President H. J. Ball was named as the Society's representative with the Assistant Secretary as alternate.

The Society has also accepted an invitation to be represented on the A.S.A. Sectional Committee on Classification of Materials for Tools, Fixtures and Gages under the sponsorship of the American Society of Tool Engineers. The appointment of two representatives is pending.

Upon nomination of the Society, Jerome Strauss, Vice-President, Vanadium Corporation of America, and a member of the Society's Executive Committee, was elected to membership on the Board of Directors of the American Standards Association for the term of three years ending December, 1943. F. M. Farmer, a Past-President of the Society, was reappointed for a term of three years, ending December, 1943, as one of the Society's representatives on the Standards Council. Past-President H. H. Morgan has been reappointed as the Society's representative on the Mechanical Standards Committee.

During the year the Society joined with the American Concrete Institute and Highway Research Board of the National Research Council in establishing a joint research project to study the durability of portland-cement concrete. A thorough study of the subject, originating with proposals at the 1939 annual meeting of the Society, established the need of basic research on the fundamental factors that affect the durability of concrete. A full account of the organization of the administrative and technical committees in charge of this work appeared in the ASTM BULLETIN for January 1941. A. T. Goldbeck and the Secretary-Treasurer represent the Society on the Administrative Committee.

The Joint Committee on Definitions of Terms Relating to Heat Treatment sponsored by the Society of Automotive Engineers, the American Society for Metals and this Society has been reconstituted in view of the need for further consideration of the subject in the light of new developments in the art and the need for effective correlation of the interest and activities of the three societies in this field. Messrs. T. S. Fuller, L. H. Fry, and H. S. Rawdon have been named as the Society's representatives on the Joint Committee, with Mr. Fuller serving as chairman of the A.S.T.M. group.

Respectfully submitted on behalf of the Executive Committee,

WM. M. BARR,
President.

C. L. WARWICK,
Secretary-Treasurer.

June, 1941.

REPORT OF COMMITTEE A-1

ON

STEEL

Among the important recommendations submitted in this report are six proposed new tentative specifications covering materials of widespread importance including low-alloy structural steel, ring and disk forgings, heat-treated wrought steel wheels, sheets in two size classifications and of utmost importance because they are basic in the forging specifications group are the proposed new requirements for blooms, billets, and slabs. New specifications have also been completed for immediate adoption as standard covering hot-worked high-carbon steel tie plates.

Three series of meetings have been held during the year: the first in Atlantic City, N. J., on June 24 and 25, 1940; the second in Philadelphia, Pa., on January 13 and 14, 1941; and the third in Washington, D. C., on March 3 and 4, 1941. Quite a number of meetings of various sections of subcommittees, particularly Subcommittee VI on Steel Forgings and Billets, have been held resulting in a total during the year of more than 40. Despite increased pressure on the time of members because of industrial activities, there has been no diminution in the intensity of Committee A-1 work. It has continued to receive the very close cooperation of a large number of its members.

Since the 1940 annual meeting, 12 new members have been elected and 6 resignations accepted, the committee membership now totaling 231, of whom 120 are classified as nonproducers and 111 as producers; of the former, 92 are consumers and 28 are general interest members. The committee is the second largest Society committee group and includes in its membership not only a large number of members who for a great many years have been associated in their daily business with the development and use of purchase specifications, but also an active group of younger men who are intimately concerned particularly with many of the newer problems in the production and use of steel.

Two New Subcommittees.—During the year two new subcommittees were organized, one on heat treatment of steel designated as Subcommittee XXIV, the other on welding of steel designated Subcommittee XXV. Lawford H. Fry, Railway Engineer, Edgewater Steel Co., accepted the chairmanship of subcommittee XXIV and Norman L. Mochel, Manager, Metallurgical Engineering, Westinghouse Electric and Manufacturing Co., is the chairman of the subcommittee on welding.

The subcommittee on heat treatment was organized because of the intensified interest in coordinating various definitions of heat treatment and also the possibility that heat treatment terms as used in various groups of steel specifications may need to be further standardized. Pending deliberations of the Joint Committee on Definitions of Terms Relating to Heat Treatment composed of representatives from the American Society for Metals, Society of Automotive Engineers, American Foundrymen's Association, and the A.S.T.M., the subcommittee is not making any specific recommendations with respect to definitions. In addition to Mr. Fry, Messrs. T. S. Fuller and H. S. Rawdon are representing the Society on the joint committee.

The subcommittee on welding of steel is organized not as a specification-writing group but as a fact-finding and coordinating committee and includes in its restricted personnel representatives of other A-1 subcommittees concerned particularly with the problem of weldability and methods of determining this property of steel, as well as other leading experts on welding. With the organization of this subcommittee the title of Subcommittee XXI which formerly read "Steel for Welding" was changed to conform to its new scope and is entitled "Steel Welding Electrodes and Welding Rods." It is now headed by J. H. Deppeler, Chief Engineer, Metal and Thermit Corp.

Subcommittee XIII on Methods of Physical Tests.—In order that there may be a clearer understanding of the activities of Subcommittee XIII on Methods of Physical Tests headed by L. H. Fry, it is deemed advisable to include in this report a statement that the subcommittee is not a standardizing group concerned with the development of specification requirements and standardized procedures for conducting tests; rather it is a fact finding and advisory body organized for the purposes of bringing to members of Committee A-1 and particularly subcommittees concerned, pertinent data on test methods, such as effect of speed of testing and others. Consequently, the personnel of the subcommittee is aimed to cover broadly the various A-1 subcommittee group interests.

Losses by Death.—During the year Committee A-1 has lost by death five members: Messrs. E. L. Lasier, U. S. Maritime Commission; J. J. Maher, Page Steel and Wire Division, American Chain and Cable Co., Inc.; C. A. McCune, Welding Engineering and Research Corp.; Robert Fairis, The Pennsylvania Railroad; and Paul E. McKinney, Bethlehem Steel Co., Inc. The committee records its sincere appreciation of their generous services. Of these five men, Mr. McKinney had been extremely active in committee work over a long period of time and because of his outstanding services to the committee the following resolution has been adopted:

RESOLUTION ON THE DEATH OF PAUL E. MCKINNEY

1885–1941

In the death on February 18, 1941, of Paul E. McKinney, Metallurgical Engineer, Bethlehem Steel Co., Inc., Committee A-1 on Steel loses one of its most active members; one who contributed much to the advancement of its development of purchase specifications for steel products. With a broad experience with specialized products for Naval armament as Superintendent of the Forge and Foundry Divisions of the U. S. Naval Gun Factory for many years, coupled with his intimate knowledge of the production of forgings, castings, and bolting material for applications at both normal and elevated temperatures, he brought to the Steel Committee an invaluable background, essential in the development of specifications.

A member of Committee A-1 since 1924 he rendered notable services on three of its subcommittees, namely, Subcommittees VI on Steel Forgings and Billets, VIII on Steel Castings, and XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service.

A list of specific activities in which his coöperation was extremely helpful would indeed be extensive When in 1933 fundamental changes were established in methods of specifying castings, his services were invaluable. When requirements for certain materials for use at extremely elevated temperatures were needed, again he assisted greatly in achieving the desired ends. Only last year when important coordination took place in standards for steel forgings, he headed an important section covering one phase of this work.

Mr. McKinney's participation in the work of Committee A-1 on Steel was marked by three outstanding characteristics. First, boundless energy—he worked hard and played hard. Too, his tenacity of purpose was always evident and whether one agreed or disagreed with him, there had to be admiration for his constant striving for the goal he believed right. And withall, no one could question his intense loyalty to Committee A-1 and its parent Society in the many activities with which he was intimately concerned.

Mr. McKinney's death is a severe loss to Committee A-1 and those groups with which he worked so closely. His many friends and associates on the committee will miss his pleasant personality and friendly spirit.

In meeting assembled, Committee A-1 on Steel pays homage to him as an outstanding metallurgical engineer and committee member and now records its appreciation for his extensive and long-time services in its behalf.

RECOMMENDATIONS AFFECTING STANDARDS AND TENTATIVE STANDARDS

As a result of work completed during the year, the committee is recommending for publication as tentative six proposed new specifications; a proposed new standard for immediate adoption; the adoption as standard of eight tentative standards; and the adoption of revisions in four standards. Also submitted are revisions in six tentative specifications and tentative revisions of eleven standard specifications.

The proposed new tentative standards and all revisions not previously published are given in detail in the Appendix, and where deemed advisable explanations of the recommended actions are given in the report, under Activities of Subcommittees. Table I lists all these recommendations and gives the results of the submission of the items to letter ballot of the 231 committee members.

ACTIVITIES OF SUBCOMMITTEES

Certain features of the committee's work are discussed below, grouped in the order of the respective subcommittees responsible.

Subcommittee I on Steel Rails and Accessories (E. F. Kenney, chairman).—Because various purchasers, including some foreign organizations, in ordering hot-worked high-carbon steel tie plates have used A.S.T.M. standards and other specifications which were not applicable to this particular material, and since the American Railway Engineering Association has specifications which are directly applicable, the subcommittee felt it desirable to recommend the immediate adoption as standard of the proposed specifications, appended hereto.[1] Essentially these follow the A.R.E.A. requirements and were redrafted in line with the A.S.T.M. editorial style.

Subcommittee II on Structural Steel for Bridges, Buildings, and Rolling Stock (F. H. Frankland, chairman).—The Standard Specifications for High-Strength Structural Rivet Steel (A 195 – 39 T) having been rather widely applied with no major criticisms resulting, the subcommittee feels justified in recommending their adoption as standard.

While the first of the so-called low alloy high tensile strength structural steels was introduced over ten years ago, the very rapid increase in the number of these steels with numerous brand names did not come about until about 1935 and the ensuing few years. With a somewhat confused situation with respect to merits of the materials, Committee A-1 and, in turn, its Subcommittee II were asked to attempt the important task of standardizing requirements. These steels are characterized by a somewhat higher tensile strength and in particular by a definitely higher yield point than the ordinary structural carbon steels. This matter was further complicated by the desirability of covering in some way the weldability of the materials. With the close cooperation of the members who recognized the necessity for clarifying the situation, the section of Subcommittee II in charge, headed by C. F. Goodrich, American Bridge Co., developed proposed specifications and the subcommittee agreed on the requirements incorporated in the proposed

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[1] These specifications were adopted as standard by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 4.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Low-Alloy Structural Steel	82	1	55
Spec. for Carbon-Steel and Alloy-Steel Ring and Disk Forgings	82	2	72
Spec. for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings	78	1	59
Spec. for Heat-Treated Wrought Steel Wheels	65	1	90
Spec. for Light Gage Structural Quality Flat Hot Rolled Carbon Steel 0.2499 and 0.1874 in. to 0.0478 in. Inclusive, in Thickness	84	3	69
Spec. for Light Gage Structural Quality Flat Rolled Carbon Steel 0.0477 in. to 0.0225 in. Inclusive, in Thickness	84	3	69
II. NEW STANDARD			
Spec. for Hot-Worked High-Carbon Steel Tie Plates	74	0	82
III. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Alloy-Steel Forgings for Locomotives and Cars (A 238 - 40 T)	79	0	77
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 40 T)	80	0	76
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 40 T)	82	0	56
Spec. for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 40 T)	76	0	62
Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 - 40 T)	86	0	52
Spec. for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 40 T)	86	0	52
IV. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Wrought Steel Wheels for Electric Railway Service (A 25 - 36)	62	1	93
Spec. for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 40; ASA B 36.3-1940)	78	1	58
Spec. for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 - 40; ASA G17.1 - 1940)	86	0	52
Spec. for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 - 40)	86	0	52
V. TENTATIVE REVISIONS OF STANDARDS			
Spec. for Steel for Bridges and Buildings (A 7 - 39; ASA G 24 - 1939)	88	1	67
Spec. for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 17 - 29); ASA G9.1 - 1933)	78	1	59
Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 40)	75	2	79
Spec. for Electric-Fusion Welded Steel Pipe (Sizes 30 in. and Over) (A 134 - 39; ASA B36.4 1939)	78	0	60
Spec. for Electric-Resistance Welded Steel Pipe (A 135 - 34; ASA B36.5-1935)	77	0	61
Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 - 39; ASA B36.9-1939)	80	0	58
Spec. for Forged or Rolled Steel Pipe Flanges for General Service (A 181 - 37)	69	0	87
Spec. for Welded and Seamless Steel Pipe (A 53 - 40; ASA B 36.1 - 1940)	86	2	68
Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 40)	77	2	77
Spec. for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 - 40)	73	0	83
Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 40)	77	0	61
VI. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for High-Strength Structural Rivet Steel (A 195 - 39 T)	100	0	56
Spec. for Hard-Drawn Steel Spring Wire (A 227 - 39 T)	77	0	79
Spec. for Steel Music Spring Wire (A 228 - 39 T)	75	0	81
Spec. for Oil-Tempered Steel Spring Wire (A 229 - 39 T)	75	0	81
Spec. for Carbon-Steel Valve Spring Quality Wire (A 230 - 39 T)	71	0	85
Spec. for Chromium-Vanadium Steel Spring Wire (A 231 - 39 T)	72	0	84
Spec. for Chromium-Vanadium Steel Valve Spring Quality Wire (A 232 - 39 T)	71	0	85
Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 - 39 T), as revised	79	0	77

tentative specifications as appended hereto.[2]

A tentative revision of the Standard Specifications for Steel for Bridges and Buildings (A 7 – 39) to provide for the deletion of references to eyebars came about because eyebars in sizes larger than 6 in. are no longer manufactured. The present requirements on bars in the specifications are deemed sufficient to cover small size eyebars.

Subcommittee VI on Steel Forgings and Billets (J. J. Laudig, chairman).—This subcommittee completed last year four new tentative specifications covering alloy and carbon steel forgings for locomotives and cars and for general industrial use (A 235, A 236, A 237, and A 238) which, with the withdrawal of certain previous specifications clarified the problem of satisfactory standards for carbon steel and alloy forgings. It recognized the desirability of covering in a separate specification carbon and alloy steel ring and disk forgings of general types manufactured. Certain ring forgings are covered in other specifications, notably those for materials for use at high temperatures, but there was no specification for what might be termed a "general" type. Consequently the proposed specifications appended hereto[3] as developed by a section headed by A. O. Schaefer, The Midvale Co., will fill this need.

One of the most difficult problems which has arisen in the steel committee's work in recent years came about in the decision to cover certain additional classes of material not provided for in the Standard Specifications for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 17 – 29). As the section in charge headed by D. R. Carse, Climax Molybdenum Co., considered the matter, it was evident that major revisions were in order involving a complete study of all types covered. Although there were widely divergent opinions on just what types should be included, what the chemistry ranges should be, and other questions, after numerous meetings and much discussion a general agreement on the revisions was reached. In this work the subcommittee had the benefit of intensive studies carried out by technical committees of the American Iron and Steel Institute on major types of steel which were in widespread usage. While the subcommittee recommends that the Standard Specifications A 17 be retained without change, they will undoubtedly be superseded by the new proposed tentative specifications appended hereto,[4] after a period of trial.

Subcommittee VII on Rolled Steel Wheels and Steel Tires (R. W. Steigerwalt, chairman).—The demand for heat-treated wrought steel wheels has increased to such a point that the producers and users of wheels believe that the A.S.T.M. should issue standards for heat-treated wheels. Subcommittee VII, after considering the matter, drafted the proposed new specifications appended hereto.[5] These are based on requirements of the Association of American Railroads, and it is believed will cover adequately this type of material.

Subcommittee VIII on Steel Castings (W. C. Hamilton, chairman).—Since the Tentative Specifications for Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 – 39 T) have been published for two years and have been in considerable use without any major criticisms, the recommendation that they be adopted as standard seemed in order. With this action the subcommittee recommends the incorporation of minor revisions involving the determination of yield point. This corresponding change will be made in three of the other specifications covering castings (A 27 – 39, A 87 - 36, and A 148 – 36) when the 1942 Book of A.S.T.M. Standards is published.

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[2] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 213.

[3] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 238.

[4] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 225.

[5] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 244.

This subcommittee has organized a new section to cooperate with Government agencies concerned with standards for steel castings so that new specifications which may be developed or revised standards will be as satisfactory as possible from the standpoint of all interests concerned. An interesting discussion of this work took place at the subcommittee's meeting in Washington.

Subcommittee IX on Steel Tubing and Pipe (T. G. Stitt, chairman).—The numerous recommendations being made by this active group, as detailed in the Appendix to this report, are for the most part self-explanatory. Generally, they are intended to bring the requirements in line with the best current industrial practices and in some cases to clarify certain inconsistencies.

The extensive changes proposed in the Standard Specifications for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 37) incorporate certain recommendations with respect to welding and also bring the specifications in line editorially with related Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 40).

The changes in Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 40) and Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 40) are related primarily to the matter of substituting schedule numbers in the method of ordering pipe.

A number of matters are being studied by the subcommittee including modifications of elongation requirements in pipe specifications which it is proposed will provide physical requirements consistent with pipe wall thicknesses. Studies of the flattening test formula and coordination of these requirements are being made. Mr. H. R. Redington, National Tube Co., is chairman of two sections responsible for these extensive studies.

An attempt to reach agreement on seamless header tube specifications resulted in definite divergence of opinion on the necessity of a separate specification; consequently, the matter has been tabled.

Subcommittee XIX on Sheet Steel and Steel Sheets (E. F. Lundeen, chairman).—For several years, Committee A-1 has been urged by numerous interests to prepare standardized requirements for steel sheets, particularly for material used in structural applications such as tank work and the like. While the committee has recognized the necessity of such requirements, and also realized the confusion created by a lack of clarity in the provisions of the structural steel specifications (A 7 – 39), which provide that flat-rolled steel in certain shapes and bars of small dimensions need not be subjected to tension tests, it has first been necessary for the manufacturers of the material to correlate considerable data and information as to their facilities and related matters. Subcommittee XIX after study of information resulting from the work of committees of manufacturers and detailed consideration of other matters including permissible variations has agreed on the two proposed specifications appended hereto.[6] It is believed these will do much to clarify the present situation and provide a large number of users of the material with satisfactory specifications.

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[6] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 218 and 222.

Subcommittee XXII on Valves, Fittings, Piping and Flanges for High-Temperature Service (A. E. White, chairman).—The majority of the numerous revisions recommended by this subcommittee effect three widely used specifications covering pipe, namely, Standard Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 40), Standard Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 - 40 T), and Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 - 40 T). Some of the modifications pertain to the question of metallographic examination and grain size, while others are intended to clarify and improve the respective sections involved.

This report has been submitted to letter ballot of the committee which consists of 231 members; 115 members returned their ballots, of whom 114 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

H. P. BIGLER,
Secretary.
R. J. PAINTER,
Staff Assistant.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee A-1 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 41 T), and

Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 41 T).

These recommendations were accepted by Committee E-10 on August 25, 1941, and assigned the A.S.T.M. serial designations indicated above. The new tentative standards appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 286 and 293.

APPENDIX

PROPOSED REVISIONS IN STANDARDS FOR STEEL

In this Appendix are given proposed revisions in specifications covering steel and steel products as referred to earlier in this report. These standards and tentative standards appear in their present form in either the 1939 Book of A.S.T.M. Standards, Part I, or the 1940 Supplement, Part I.

NEW STANDARD

The proposed Standard Specifications for Hot-Worked High-Carbon Steel Tie Plates, appended hereto,[1] are recommended for immediate adoption as standard and the committee accordingly asks for a favorable nine-tenths vote in order that this recommendation may be referred to letter ballot of the Society.

REVISIONS OF TENTATIVE STANDARDS

Tentative Specifications for Alloy-Steel Forgings for Locomotives and Cars (A 238 – 40 T):

Table I.—Change the requirements for classes B and C forgings to read as prescribed in the accompanying Table I by the addition of the italicized figures:

TABLE I.—TENSILE REQUIREMENTS FOR CLASSES B AND C ALLOY-STEEL FORGINGS.

Class	Size, Solid Diameter or Thickness, in. Over	Size, Solid Diameter or Thickness, in. Not Over	Tensile Strength, min., psi.	Yield Point, min., psi.	Elongation in 2 in., min., per cent	Reduction of Area, min., per cent
B (Normalized and Tempered)	..	5	90 000	60 000	24	48
	5	9	90 000	60 000	22	44
	9	*13*	90 000	58 000	21	42
	13	20	*88 000*	*56 000*	*20*	*40*
C (Normalized and Tempered)	..	5	95 000	72 000	23	55
	5	9	95 000	70 000	22	53
	9	*13*	93 000	67 000	22	50
	13	20	*91 000*	*65 000*	*21*	*48*

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 40 T):

Section 1 (a).—Change the phrase "two grades" to read "three grades."

Section 4 (a).—In the tabulation of chemical requirements change the carbon content of Grade T1 from "0.08 to 0.20" per cent to read "0.10 to 0.20" per cent.

Add chemical composition requirements for a new grade designated as T1b, to read as follows:

	Grade T1b
Carbon, per cent	0.08 to 0.14
Manganese, per cent	0.30 to 0.60
Phosphorus, max., per cent	0.04
Sulfur, max., per cent	0.05
Silicon, per cent	0.10 to 0.50
Molybdenum, per cent	0.45 to 0.65

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[1] These specifications were adopted as standard by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 4.

Section 6 (*a*).—Add tensile requirements for a new grade designated as T1b, to read as follows:

	Grade T1b
Tensile strength, min., psi..........	53 000
Yield point, min., psi.............	28 000
Elongation in 2 in., min., per cent..	30

Section 9 (*c*).—For the new grade T1b add requirements for Brinell hardness and Rockwell hardness numbers the same as for grade T1, namely, 137 and B77, respectively.

Section 19 (*a*).—Change to read as follows by the addition of the italicized symbol:

19. (*a*) The name or brand of the manufacturer, the grade of material from which it is made (T1 or T1a or *T1b*), A.S.T.M. Specifications A 209, together with the hydrostatic test pressure in pounds per square inch, and whether hot-finished or cold-drawn, shall be legibly stenciled on each tube 1¼ in. in outside diameter and over, provided the length is not under 3 ft.

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 40 T):

Section 2 (*d*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*d*) *When requested by the purchaser and so stated in the order*, the steel melting practice shall be such as will produce a structural grain size in a *proof test* specimen of the pipe corresponding to an A.S.T.M. grain size of from 3 to 6 if given the following specific heat treatment: heating to 1700 F. and holding approximately 1 hr. per inch of wall thickness, but in no case less than 1 hr. [and cooling in the furnace at a rate of not to exceed 50 deg. Fahr. per hour until below 1000 F.] *and cooling in still air to below 1000 F.*

Section 2 (*e*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*e*) *Where the steel melting practice specified in Paragraph* (*d*) *is required*, hot-finished pipe in the as-finished condition in nominal sizes 3 in. and over made to schedules 140 and 160 shall [conform to the grain size requirements specified in Paragraph (*d*)] *have an A.S.T.M. grain size of from 3 to 6.*

Section 7 (*a*).—Change the last line of the table from its present form: namely,

9 and over............ 100 or fraction thereof

to read as follows:

6 and over............ 100 or fraction thereof

Section 11.—Change the Note to read as follows by the addition of the italicized words and the omission of those in brackets:

NOTE.—When requested by the purchaser and so stated in the order, pipe in sizes 14 in. in nominal diameter and smaller shall be tested to one and one-half times the specified working pressure [(where one and one-half times the specified working pressure exceeds 2800 psi.)], provided the fiber stress corresponding to those test pressures does not exceed one half the minimum specified yield point of the material, as determined by the above formula. *When one and one-half times the working pressure exceeds 2800 psi. the hydrostatic test pressure shall be a matter of agreement between the purchaser and the manufacturer.*

Section 12.—Change from its present form to read as follows:

12. The manufacturer shall furnish two photomicrographs at 100 diameters for each individual size and wall thickness rolled from each melt taken as follows: (*1*) one of a specimen of pipe in the as-finished condition, and (*2*) one of a proof test specimen of pipe which has been heated to 1700 F., held approximately 1 hr. per in. of wall thickness, but in no case less than 1 hr., and normalized by cooling in still air until below 1000 F. When requested by the purchaser and so stated in the order, the grain size shall conform to the requirements of Section 2 (*d*) and (*e*), respectively. Such photomicrographs shall be suitably identified as to pipe size, wall thickness, and melt. No photomicrographs for the individual pieces purchased shall be required except as specified in Paragraph S7.

Section 21.—Change the first sentence to read as follows by the addition of the italicized words:

> Each length of pipe manufactured in accordance with these specifications shall be legibly marked, either by stenciling, stamping, or rolling, with the manufacturer's private identifying mark, with the designation A 206, and an additional symbol S if the pipe conforms to the supplementary requirements specified in Paragraphs S1 to S7, the length, the ASA schedule number, ***the hydrostatic test pressure,*** and the heat number or manufacturer's number by which the heat can be identified.

Paragraph S5.—Change from its present form to read as follows:

> S5. The steel shall have a homogeneous structure as shown by the etching tests described in Paragraph S6. When requested by the purchaser and so stated in the order, the grain size of the steel shall conform to the requirements of Section 2 (*d*) and (*e*), respectively.

Paragraph S6.—Delete the second sentence of this paragraph: namely,

> Sections used for the flattening test described in Section 10 may be used for the etching test.

Paragraph S7.—Change from its present form to read as follows:

> S7. In addition to the photomicrographs required in Section 12, the purchaser may specify that photomicrographs shall be furnished from each end of one or more pieces from each lot of pipe in the as-finished condition. The purchaser shall state in the order the number of pipes to be tested from each lot. When photomicrographs are required on each piece, the photomicrographs from each lot of pipe in the as-finished condition required in Section 12 may be omitted, but the proof test photomicrographs shall be furnished as specified in Section 12. When requested by the purchaser and so stated in the order, the grain size shall conform to the requirements of Section 2 (*d*) and (*e*), respectively. All photomicrographs required shall be properly identified as to heat number, size, and wall thickness of pipe from which the section was taken. Photomicrographs shall be further identified to permit association of each photomicrograph with the individual piece of pipe it represents.

Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 40 T):

Table I.—Change the manganese content for grade P 5b (4 to 6 per cent chromium-silicon-molybdenum) from "0.30 max." to read "0.50 max." per cent. Change the carbon content of grade P 5c (4 to 6 per cent chromium-molybdenum stabilized with titanium or columbium) from "0.10 or, as specified, 0.15 max.," to read "0.12 max." and the titanium content from its present value of "4 to 6 times carbon," to read "min. per cent, 4 times the actual carbon; max., 0.70 per cent." Change the manganese content of grade P 15 (silicon-molybdenum) from "0.30 max." to "0.30 to 0.60" per cent.

Section 11.—Make the same change in this Note as recommended above in Section 11 of Specifications A 206 – 40 T.

Paragraph S6.—Make the same change in this paragraph as recommended above in Paragraph S6 of Specifications A 206 – 40 T.

Section 20.—Change the first sentence to read as follows by the addition of the italicized words and the omission of the word in brackets:

> Each length of pipe manufactured in accordance with these specifications shall be legibly marked, either by stenciling, stamping, or rolling, with the manufacturer's private identifying mark, with the [symbols] *designation* A 158, ***the length, the hydrostatic test pressure, the type composition,*** and an additional symbol S if the pipe conforms to the supplementary requirements specified in Paragraphs S1 to S6.

Tentative Specifications for Carbon-Steel Castings Suitable for Fusion Welding for Service at Temperatures up to 850 F. (A 216 – 40 T):

Section 1.—Delete the last two sentences in Note 2 and add the following:

> Grade WCB is applicable to the design standards formulated by the Sectional Committee on Standardization of Pipe Flanges and Fittings, under the procedure of the American Standards Association, as given in the American Standard for Steel Pipe Flanges and Flanged Fittings (ASA No.: B16*e*-1939). The Sectional Committee is now developing pressure ratings for the other grade of material covered in this specification.

Section 16.—Delete the Note and add a new Paragraph (*b*) to read as follows:

> (*b*) Finished products shall be marked in accordance with the Standard Marking System for Valves, Fittings, Flanges, and Unions (No. SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.

Tentative Specifications for Alloy-Steel Castings Suitable for Fusion Welding for Service at Temperatures from 750 to 1100 F. (A 217 - 40 T):

Section 1.—Delete the second paragraph of Note 2 and add the following:

> Grades WC1 and WC4 are applicable to the design standards formulated by the Sectional Committee on Standardization of Pipe Flanges and Fittings, under the procedure of the American Standards Association, as given in the American Standard for Steel Pipe Flanges and Flanged Fittings (ASA No.: B16*e*-1939). The Sectional Committee is now developing pressure ratings for the other grades of materials covered in this specification.

Section 17.—Make the same change in this section as recommended in Section 16 of Specifications A 216 - 40 T.

Revisions of Standards, Immediate Adoption

Standard Specifications for Wrought Steel Wheels for Electric Railway Service (A 25 - 36):

Section 8.—Change Paragraph (*j*) from its present form: namely,

> (*j*) *Diameter of Hub.*—The diameter of hub shall not be less but may be ¾ in. more than that specified. The thickness of wall of the finished bored hub, unless otherwise specified, shall not be less than 1 in. at any point for bores 6 in. or under in diameter, nor less than 1¼ in. for bores over 6 in. in diameter. The thickness of the hub wall in any wheel shall not vary more than ⅜ in. at any two points equidistant from the face of the hub.

to read as follows:

> (*j*) *Diameter of Hub.*—Unless otherwise specified, the thickness of wall of finished bored hub shall be not less than 1 in. at any point for bores 6 in. and under in diameter, nor less than 1¼ in. for bores over 6 in. in diameter. The outside diameter shall not exceed that specified by more than ¾ in. The thickness of the hub wall in any wheel shall not vary more than ⅜ in. at any two points equidistant from the face of the hub.

Standard Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 40):

Table I.—Change the manganese content of grade H (seamless) from "0.30 to 0.60" to read "0.30 to 0.90" per cent.

Section 8.—Make the same change in this Note as recommended above in Section 11 of Specifications A 206 - 40 T.

Section 17.—Change the first sentence to read as follows by the addition of the italicized words:

> Each length of pipe manufactured in accordance with these specifications shall be legibly marked, either by stenciling, stamping, or rolling, with the manufacturer's private identifying mark, together with the symbols A 106-A (or B depending on the grade of seamless steel used, or no letter if lap-welded), *the hydrostatic test pressure,* and an additional S if the pipe conforms to the supplementary requirements specified in Paragraphs S1 to S6.

Paragraph S6.—Delete the second sentence of this paragraph: namely,

> Sections used for the flattening tests specified in Section 7 (*b*) and (*c*) may be used for the etching test.

Standard Specifications for Alloy-Steel Castings for Valves, Flanges, and Fittings for Service at Temperatures from 750 to 1100 F. (A 157 - 40):

Table I.—Add a new grade C 12 covering an 8 to 10 per cent chromium-molybdenum steel with the following chemical requirements:

	Grade C 12
Carbon, max., per cent	0.30
Manganese, max., per cent	1.00
Phosphorus, max., per cent	0.05
Sulfur, max., per cent	0.06
Silicon, max., per cent	1.00
Chromium, per cent	8.00 to 10.00
Molybdenum, per cent	1.10 to 1.50

Table II.—Change the minimum tensile strength requirement of grades C 3A and C 3B from "100,000" to read "90,000" psi.; also, change the minimum yield point requirement of these grades from "65,000" to "60,000" psi. Add new physical requirements for the new grade C 12 to read as follows:

	Grade C 12
Tensile strength, min., psi.	90 000
Yield point, min., psi.	60 000
Elongation in 2 in., min., per cent	18
Reduction of area, min., per cent	30

Section 17.—Delete the Note and add a new Paragraph (*b*) to read as follows, relettering present Paragraph (*b*) as (*c*):

> (*b*) Finished products shall be marked in accordance with the Standard Marking System for Valves, Fittings, Flanges, and Unions (No. SP-25-1936) of the Manufacturers Standardization Society of the Valve and Fittings Industry.

Standard Specifications for Carbon-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 95 – 40):

Section 16.—Make the same change in this section as recommended in Section 17 of Specifications A 157 – 40.

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Steel for Bridges and Buildings (A 7 – 39):

Section 9.—In Paragraph (*a*) change the text to read as follows by the addition of the word in italics and the omission of those in brackets:

> The material, except as specified in Sections 3 and 4 and [Paragraphs (*b*) and] ***Paragraph*** (*d*) of this section, shall conform to the following requirements as to tensile properties:

Delete all the requirements for eyebar flats, unannealed, and footnote *a* which reads as follows: "These requirements apply when full-size tests of annealed eyebars are not specified. See Paragraph (*b*)."

Delete Paragraphs (*b*) and (*c*).

Change Paragraph (*f*) to read as follows by the omission of the words in brackets:

> (*f*) For material over $\frac{3}{4}$ in. in thickness or diameter, a deduction from the percentage of elongation in 8 in. specified in Paragraph (*a*) of 0.25 per cent shall be made for each increase of $\frac{1}{32}$ in. of the specified thickness or diameter above $\frac{3}{4}$ in. to a minimum of 18 per cent for plates, shapes, and bars [and a minimum of 14 per cent for eyebar flats unannealed].

Section 10.—In the tabulation of requirements for the ratio of pin diameter to thickness of specimen, delete all those for eyebar flats, unannealed.

Section 11 (*b*).—Change to read as follows by the omission of the words in brackets:

> (*b*) Test specimens for annealed material [except eyebars,] shall be prepared from the material as annealed for use or from a short length of a full section from the same melt similarly treated.

Standard Specifications for Carbon-Steel and Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 17 – 29):

These revisions are in the form of proposed new tentative specifications, appended hereto[2] which are intended to replace when adopted, the present standard.

Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 40):

Section 3 (*b*).—Add a footnote after

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[2] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 225.

the word "composition" to read as follows:

> If desired, alternate type B (open-hearth iron) may be specified conforming to the chemical requirements given above. This alternate type B shall meet all the tests and other requirements now specified for type B.

Add chemical composition requirements for a new type of material to be designated as alternate type B, open-hearth iron, as follows:

	Alternate Type B
Carbon, max., per cent..........	0.05
Manganese, max., per cent.......	0.35
Phosphorus, max., per cent.......	0.02
Sulfur, max., per cent............	0.045
Molybdenum, per cent...........	0.05 to 0.15
Copper, min., per cent...........	0.40

Add a footnote after the words "type A" to read as follows:

> When Type A tubes are specified with copper content, the copper range shall be 0.20 to 0.35 per cent.

Standard Specifications for Electric-Fusion Welded Steel Pipe (Sizes 30 in. and Over) (A 134 – 39):

Section 1.—Change the second sentence to read as follows by the omission of the words and figures in brackets:

> The pipe is intended for conveying liquid, gas, or vapor [at temperatures below 450 F.].

Standard Specifications for Electric-Resistance-Welded Steel Pipe (A 135 – 34):

Section 1.—Change to read as follows by the omission of the words in brackets:

> 1. These specifications cover two grades of electric-resistance-welded steel pipe 30 in. and under in diameter. The pipe is intended for conveying liquid, gas, or vapor [at temperatures below 450 F.]; and only grade A is adapted for flanging and bending. The suitability of pipe for various purposes is somewhat dependent upon its dimensions, properties, and conditions of service, so that the purpose for which the pipe is intended should be stated in the order.

Section 14 (a).—Change the requirement on the pipe bevel from its present figure of "45 deg." to read "37.5 deg." Throughout this section change the figure "45 deg." to read "37.5 deg."

Standard Specifications for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 39):

Section 1.—Make the same change in this section as recommended above in Section 1 of Specifications A 135 – 34.

Section 14 (a).—Make the same change in this section as recommended above in Section 14 (a) of Specifications A 135 – 34.

Standard Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40):

Table I.—Change the carbon content of grade T 16 (chromium-molybdenum-titanium) from "0.10 max." to "0.12 max." and change the titanium content from "0.35 to 0.60" to read "min. 4 times actual carbon; max. 0.70."

Standard Specifications for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 37):

Classes.—Throughout these specifications change the word "classes" to read "grades."

Section 4.—Change the title of this section from "Grain Flow" to "Manufacturing Practice." Delete the present Paragraph (*a*) and add the new Paragraphs (*a*), (*b*), (*c*), and (*d*) listed below, relettering the present Paragraph (*b*) as (*e*):

> (*a*) Material for flanges shall consist of blooms, billets, slabs, or bars either forged or rolled from an ingot, and cut to the required length by a process that will not produce injurious defects in the flange.
>
> (*b*) Forging or rolling operations may be performed either by hammering, pressing, rolling, extruding, or upsetting. Flanges shall be

brought as nearly as practicable to the finished shape and size by hot working and shall be so processed as to cause metal flow during the hot-working operation in the direction most favorable for resisting the stresses encountered in service.

(*c*) When specified in the order, a sample flange may be sectioned and etched to show flow lines and the condition as regards internal imperfections. In such cases, the question of acceptable and unacceptable character of metal flow shall be a subject for agreement between the manufacturer and the purchaser.

(*d*) When specified in the order, the manufacturer shall submit for approval of the purchaser a sketch showing the shape of the rough forging before machining.

Section 5.—Change from its present form to read as follows:

5. The steel shall conform to the following requirements as to chemical composition:

	Grade I	Grade II
Carbon, max., per cent.....	0.35[a]	[a]
Manganese, max., per cent..	0.90	0.90
Phosphorus, max., per cent .	0.05[b]	0.05[b]
Sulfur, max., per cent.......	0.05[b]	0.05[b]

[a] The carbon content of grade II material shall be a matter of agreement between the manufacturer and the purchaser. When flanges will be subject to fusion welding, the carbon content shall not exceed 0.35 per cent. When the carbon is restricted to 0.35 per cent, max., it may be necessary to add silicon to the composition for grade II and for the heavier thicknesses of grade I flanges in order to meet the required tensile properties. The silicon content shall not exceed 0.30 per cent.

[b] See Section 7.

Section 9.—Change from its present form to read as follows:

9. (*a*) The tension test specimens taken from the flanges shall be machined to the form and dimensions shown in Fig. 1, except as specified in Paragraph (*b*).

(*b*) In the case of small sections which will not permit of taking the standard test specimen specified in Paragraph (*a*), the tension test specimens shall be as large as feasible and its dimensions shall be proportional to those shown in Fig. 1. The gage length for measuring elongation shall be four times the diameter of the specimen.[a]

(*c*) For the purpose of making tension tests the manufacturer shall supply suitable test specimens from the flange or, at his option, furnish test blanks prepared from the billets or forging bar entering into the finished product, provided such test blank has undergone approximately the same working and heat treatment as the finished product.

Section 14.—Change from its present form to read as follows:

14. Identification marks shall be legibly stamped on each forging.

Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 40):

Table III.—Change from its present form to read as prescribed in the accompanying Table II.

Appendix.—In the table, delete the values for nominal wall thicknesses for the following sizes of pipe: 20 in., Schedule Number 160; 24 in., Schedule Numbers 140 and 160; 30 in., Schedule Numbers 10, 20, and 30.

Change the first sentence in the introductory paragraph of the Appendix to read as follows by the addition of the italicized words and the omission of those in brackets:

Except for deletion of thicknesses for the 20 and 24 in. sizes in Schedule 160, the 24 in. size in Schedule 140, and all 30 in. sizes which are not commercially available, the following table is identical with [The following is a reprint of] Table 2 of the American Standard for Wrought-Iron and Wrought-Steel Pipe (ASA No.: B36.10—1939) of the American Standards Association which is referred to in the Note under Section 12 (*a*) of these specifications. *This table is published here for information only.*

Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 40):

Table II.—Change from its present form to read as prescribed in the accompanying Table II.

Appendix.—Delete the entire Appendix and Table.

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[a] These requirements are in accordance with the provisions of Section 7 of the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials, 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 453.

TABLE II.—STANDARD WEIGHTS AND DIMENSIONS OF WELDED AND SEAMLESS STEEL PIPE.[a]

NOTE 1.—Where more than one weight is listed under the same size and class, the order should definitely specify the wall thickness desired.

Size (Nominal Inside Diameter), in.	Outside Diameter, in.	Number of Threads per Inch	"Standard Weight" Pipe				"Extra Strong" Pipe				"Double Extra Strong"[b] Pipe	
			Schedule 30		Schedule 40		Schedule 60		Schedule 80			
			Thickness, in.	Weight of Pipe per Linear Foot, Threaded and with Couplings, lb.	Thickness, in.	Weight of Pipe per Linear Foot, Threaded and with Couplings, lb.	Thickness, in.	Weight of Pipe per Linear Foot, Plain Ends, lb.	Thickness, in.	Weight of Pipe per Linear Foot, Plain Ends, lb.	Thickness, in.	Weight of Pipe per Linear Foot, Plain Ends, lb.
⅛	0.405	27	....	....	0.068	0.25	....	....	0.095	0.31	....	....
¼	0.540	18	....	....	0.088	0.43	....	....	0.119	0.54	....	....
⅜	0.675	18	....	....	0.091	0.57	....	....	0.126	0.74	....	....
½	0.840	14	....	....	0.109	0.85	....	....	0.147	1.09	0.294	1.71
¾	1.050	14	....	....	0.113	1.13	....	....	0.154	1.47	0.308	2.44
1	1.315	11½	....	....	0.133	1.68	....	....	0.179	2.17	0.358	3.66
1¼	1.660	11½	....	....	0.140	2.28	....	....	0.191	3.00	0.382	5.21
1½	1.900	11½	....	....	0.145	2.73	....	....	0.200	3.63	0.400	6.41
2	2.375	11½	....	....	0.154	3.68	....	....	0.218	5.02	0.436	9.03
2½	2.875	8	....	....	0.203	5.82	....	....	0.276	7.66	0.552	13.70
3	3.500	8	....	....	0.216	7.62	....	....	0.300	10.25	0.600	18.58
3½	4.000	8	....	....	0.226	9.20	....	....	0.318	12.51	0.636	22.85
4	4.500	8	....	....	0.237	10.89	....	....	0.337	14.98	0.674	27.54
5	5.563	8	....	....	0.258	14.81	....	....	0.375	20.78	0.750	38.55
6	6.625	8	....	....	0.280	19.19	....	....	0.432	28.57	0.864	53.16
8	8.625	8	0.277	25.00	0.322	28.81	....	....	0.500	43.39	0.875	72.42
*10	10.750	8	0.307	35.00	0.365	41.13	0.500	54.74	....	....	....	....
**12	12.750	8	0.330	45.00	**0.375	50.71	**0.500	65.41	....	....	....	....

[a] Sizes larger than those shown in the table are measured by their outside diameter, such as 14 in. in outside diameter, 16 in. in outside diameter, etc.

These larger sizes will be furnished with plain ends, unless otherwise specified.

The weights will correspond to the manufacturers' published standards although it is possible to calculate the theoretical weights for any given size and wall thickness on the basis of 1 cu. in. of steel weighing 0.2833 lb.

[b] The American Standard for Wrought-Iron and Wrought-Steel Pipe (ASA B36.10-1939) has assigned no schedule number to "Double Extra Strong" pipe.

* Standard weight pipe 10-in. in nominal size is also available with 0.279 in. wall thickness, but this wall is not covered by a schedule number.

** Owing to a departure from the "Standard Weight" and "Extra Strong" wall thicknesses for the 12-in. nominal size, Schedules 40 and 60, in Table 2 of the American Standard for Wrought-Iron and Wrought-Steel Pipe (ASA B36.10-1939) the regular "Standard" and "Extra Strong" wall thicknesses (0.375 in. and 0.500 in.) have been substituted.

Standard Specifications for Seamless Cold-Drawn Alloy-Steel (4 to 6 per cent Chromium) Heat-Exchanger and Condenser Tubes (A 187 – 40):

Section 12 (*d*).—Change from its present form: namely,

(*d*) From each heat-treated lot, one flattening test and one flaring or flange test specified in Sections 7 and 8 shall be made on each of two tubes, and one hardness test specified in Section 9 shall be made on each of 5 per cent of the tubes.

to read as follows:

(*d*) From each heat-treated lot, one flattening test and one flaring or flange test specified in Sections 7 and 8 shall be made on each of two tubes, and one hardness test specified in Section 9 shall be made on each of 5 per cent of the tubes. For the purpose of calculating the number of tests on tubes heat treated by the continuous process, the number of tubes in a heat-treated lot shall be determined from the size of the tubes as follows:

Size of Tube	Size of Lot
Less than 2 in. but over 1 in. in outside diameter or over 1 in. in outside diameter and under 0.200 in. in wall thickness	not more than 75 tubes
1 in. or less in outside diameter	not more than 125 tubes

ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the following tentative standard be approved for reference to letter ballot of the Society for adoption as standard with the revision as indicated below:

Tentative Specifications for Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 – 39 T):

Section 8 (c).—Change to read as follows by the addition of the italicized words:

(*c*) The yield point shall be determined by the drop of the beam or halt in the gage of the testing machine, *or by the use of dividers*, or by an extensometer, at a crosshead speed not to exceed ⅛ in. per min. *Where a definite yield point is not exhibited, the yield strength corresponding to a limiting permanent offset of 0.2 per cent of the gage length of the specimen shall be used instead. The "offset method" of determining yield strength as described in the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials shall be followed.* The tensile strength shall be determined at a speed of head not to exceed 1½ in. per min.

EDITORIAL CHANGES IN STANDARDS

Tentative Specifications for Carbon-Steel Forgings for Locomotives and Cars (A 236 – 40 T):

Section 13 (c).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

(*c*) For special normalized and tempered forgings, one tension test and, if specified, [one etch test for microscopic examination] *one microscopic test from an etched specimen* shall be made from each class by size of each melt in each heat treatment lot. For information only, when specified, [one unetched test for microscopic examination] *one microscopic test from an unetched specimen* shall be made from the largest size forging in each melt.

Tentative Specifications for Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings (A 234 – 40 T):

Section 1 (a).—Change to read as follows by the addition of the italicized words:

(*a*) These specifications cover factory-made wrought carbon-steel and carbon-molybdenum-steel welding fittings for pressure piping. The term "welding fittings" applies to butt-welding or socket-end parts such as 45-deg. and 90-deg. elbows, 180-deg. return bends, caps, tees, reducers, lapped joint stub ends, and other types *as covered by the American Standards for Steel Butt-Welding Fittings (ASA B16.9-1940) and for Steel Socket-Welding Fittings (ASA B16.11-1941)*.

Standard Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40):

Table I.—To correct a typographical error in the present printing, change the silicon content requirement for grade T 13 (Chromium-Silicon-Molybdenum) from "1.00 to 1.60" to read "1.00 to 2.00" per cent.

Standard Specifications for Commercial Cold-Finished Bar Steels and Cold-Finished Shafting (A 108 – 36):

Table III.—Change the permissible variations for hexagons, to read as follows by the addition of the italicized figures and the omission of those in brackets:

	Variations from Size, in.			
	Carbon 0.30 per cent and less		Carbon 0.31 to 0.50 per cent	
	Under	Over	Under	Over
Specified Dimensions Between Opposite Sides, in.:				
[0.3] 5/16 and under	0.002	0	0.003	0
Over [0.3] 5/16 to 1, incl.	0.003	0	0.004	0
Over 1 to 2½, incl.	0.004	0	0.005	0
[Over 2½ to 4, incl.] *Over 2½ to 3 9/16, incl.*	0.005	0	0.006	0
Over 3 9/16 to 4, incl.	[0.005] *0.006*	0	[0.006] *0.007*	0

In the first column of the table under "Squares" change "0.3" to read "$\frac{5}{16}$" in two places.

In the first column of the table under "Flats" change "0.3" to read "$\frac{5}{16}$" in four places. For flat bars with carbon 0.30 per cent and less, change the permissible variations in width and thickness from "0.003 in., 0.004 in., and 0.005 in." to read "0.002 in., 0.003 in., and 0.004 in." respectively for the several thicknesses specified for widths less than $1\frac{1}{2}$ in.

REPORT OF COMMITTEE A-3

ON

CAST IRON

Since the 1940 annual meeting of the Society, Committee A-3 on Cast Iron has held one meeting, in Washington, D. C., on March 4, 1941. A second meeting will be held in June during the annual meeting of the Society.

It is with sincere regret that the committee records the death of Mr. Paul E. McKinney, Metallurgical Engineer, The Bethlehem Steel Co. At its March meeting the following resolution on the untimely passing of Mr. McKinney was adopted by the committee:

> RESOLVED: That in the death of Mr. P. E. McKinney, the committee has lost the advice and the assistance of a metallurgical engineer whose recognized ability was of great value to the committee and whose zeal in committee work was outstanding. The committee will miss his help and companionship, and expresses its sympathy to his survivors.

Since the 1940 annual meeting, the following seven new members have been elected:

- The Cooper-Bessemer Co., represented by T. E. Egan,
- Richard Schneidewind, University of Michigan,
- Tinius Olsen Testing Machine Co., represented by B. L. Lewis,
- U. S. War Dept., represented by W. F. Tompkins with Granville Smith, alternate,
- U. S. Army Ordnance Dept., Watertown Arsenal, represented by S. B. Ritchie,
- U. S. Bureau of Ships, represented by E. C. Forsyth, and
- Cornell University, Dept. of Chemistry, represented by C. W. Mason.

In addition, A. P. Spooner now represents the Bethlehem Steel Co. in place of P. E. McKinney, deceased.

The total membership of the committee is now 113, of whom 36 are classified as producers, 47 as consumers, and 30 as general interest members.

In accordance with the instructions of Committee A-3, the chairman met with representatives of the Gray Iron Division of the American Foundrymen's Association in New York City on May 14, 1941, and plans for educational work for promoting utilization and support for formulation of A.S.T.M. specifications were discussed. These will be reported at the June meeting of the committee.

It was the consensus of opinion of the committee at its meeting in Washington on March 4 that it is the policy of the Society to take the lead in specification problems, and in this emergency period it would be highly desirable that all specifications for cast iron be correlated, clarified, and simplified wherever possible. The A.S.T.M. Standard Specifications for Gray-Iron Castings (A 48 – 36) are backed by much research and study, and they permit selection of cast iron according to tensile strength classes and according to the sections in which the castings may be used. These and other A.S.T.M. specifications, have not always been correctly adapted and interpreted when used as bases for other specifications. It was called to the attention of Committee A-3 that there is within the foundry trade some confusion and hence antipathy toward specifications because of the large number, and sometimes confusing provisions, of the specifications.

I. NEW TENTATIVE STANDARD

On the recommendation of Subcommittee VII on Microstructure of Cast Iron, Committee A-3 recommends that the new proposed Tentative Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron be accepted for publication as tentative as appended hereto.[1]

II. ADOPTION AS STANDARD OF TENTATIVE STANDARD

The committee recommends, as proposed by Subcommittee XXI on Pressure Pipe, that the Tentative Specifications for Cast Iron Pit-Cast Pipe for Water and Other Liquids (A 44 - 39 T)[2] be approved, without revision, for reference to letter ballot of the Society for adoption as standard.

III. REVISIONS OF STANDARDS

On the recommendation of Subcommittee VI on General Castings, the committee recommends the following addition to the Standard Specifications for Gray Iron Castings (A 48 - 36)[3] and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society:

Table I.—For Class No. 60 iron add a minimum breaking load requirement of 12,500 lb. in the transverse or flexure test on 2.0 in. diameter bar (Type C) tested on 24-in. supports.

On the recommendation of Subcommittee XVIII on Automotive Castings, the committee recommends for immediate adoption the following revision in the Standard Specifications for Automotive Gray-Iron Castings (A 159 - 40)[4] and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Table III.—Change the requirement for total carbon for alloy No. 112 from "3.45 ±0.15 per cent" to "3.55 ± 0.15 per cent" and change the requirement for silicon for the same alloy from "2.90 ± 0.10 per cent" to "2.75 ± 0.15 per cent."

Section 8 (*b*).—Change the last sentence of this paragraph to read as follows by the addition of the italicized words and the omission of those in brackets:

> In general, one or more of the elements chromium, copper, molybdenum, *vanadium*, nickel, and *titanium* [should] *may* be used.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 113 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron	64	0	4
II. ADOPTION AS STANDARD OF TENTATIVE STANDARD			
Spec. for Cast-Iron Pit-Cast Pipe for Water and Other Liquids (A 44 - 39 T)	47	0	13
III. REVISIONS OF STANDARDS			
Spec. for Gray-Iron Castings (A 48 - 36), immediate adoption	59	1	8
Spec. for Automotive Gray-Iron Castings (A 159 - 40), immediate adoption	46	0	14

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Pig Iron (H. M. Rich, chairman) has in preparation new specifications for pig iron which are intended as a revision of and to replace the Standard Specifications for Foundry Pig Iron (A 43 - 24). In this connection the Federal Specification for Foundry Pig Iron (QQ-I-676) and a proposed American Iron and Steel Institute specification are being reviewed to ascertain to what extent correlation of these specifications can be made.

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[1] This recommended practice was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 300.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 960.

[3] 1939 Book of A.S.T.M. Standards, Part I, p. 482.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 143.

Subcommittee III on Cylinders (O. B. Schultz, chairman) is giving consideration to a revision of the Standard Specifications for Cast-Iron Locomotive Cylinders (A 45 – 14). The present specifications are obsolete and will be either completely revised or withdrawn.

Subcommittee V on Light Gray-Iron Castings (W. J. Grede, chairman) prepared the Specifications for Lightweight and Thin-Sectioned Gray-Iron Castings (A 190 – 40) which were adopted as standard in 1940. Since the work of the subcommittee has been completed, upon the request of the chairman, Subcommittee V has been discharged with thanks for its prompt and thorough work. Mr. W. J. Grede will act in an advisory capacity to Committee A-3 in matters pertaining to Specifications A 190.

Subcommittee VI on General Castings (H. Bornstein, chairman) recommends the revision given earlier in this report and also presents as an editorial change the following explanatory clauses on deflection values to be added to the Standard Specifications for Gray Iron Castings (A 48 – 36):[4]

Note 4.—Add the following at the end of this note:

> Deflection values have been omitted from these specifications because the desired deflection would be governed by the use of the iron. In some cases a maximum value would be specified because a stiff iron is desired and in other cases minimum values would have to be specified because a flexible iron is required. Consequently the deflection figures should be reached by agreement between the manufacturer and the purchaser.
>
> The use of modulus of elasticity gives a definite load-deflection relationship which is superior to a specification for deflection unless that specification provides for increase in deflection with increase in load. The maximum modulus of elasticity could be specified if toughness is desired or a minimum value could be specified if stiffness is desired. For the 1.20-in. diameter test bar the formula for modulus of elasticity is as follows:
>
> $$\text{Modulus of elasticity, psi.} = \frac{\text{Load in pounds}}{\text{Deflection in inches}} \times 1194$$

The subcommittee has called to the attention of Committee A-3 the fact that in the case of Class 70 of the Federal Specification for Gray-Iron Castings (QQ-I-652) no tensile requirements are given or specified and this may be confusing since for other classes, which correspond to the classes prescribed in Specifications A 48, definite minimum tensile strength requirements are specified.

Subcommittee VI is studying the U. S. Navy Specifications for Gray-Iron Castings (46 I 6c) and for High-Test (Semisteel) Gray Iron Castings (46 I 5b) and will report at the June meeting of the committee.

The subcommittee is cooperating with Subcommittee XVII in attempting to arrive at a degree of correlation between Specifications A 48 and the Standard Specifications for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 – 40). A joint report and recommendation will be presented at the June meeting of the committee.

The Proposed Specifications for Manhole Frames and Covers that are being prepared by Sectional Committee A 35 on Manhole Frames and Covers have been reviewed in draft form and found in general to be compatible with A.S.T.M. Specifications A 48. Several minor suggested changes have been proposed. Material Specification No. 110 of the Hydraulic Institute has also been reviewed and found to be in general agreement with Specifications A 48.

Specification S-13 for Gray Iron Castings appearing in the A.S.M.E. Boiler Construction Code is identical with A.S.T.M. Specifications A 48 as concerns the requirements for classes 20, 30, and 40. Action has recently been taken by the Boiler Code Committee to add requirements for two additional classes, 35 and 45, so that the Code Specification will now cover five classes identical with Standard A 48.

Subcommittee VII on Microstructure of Cast Iron (G. F. Comstock, chairman) has completed its work in cooperation with the A.F.A. Committee on Classification of Graphite in Gray Iron (W. E. Mahin, chairman) in the preparation of a Proposed Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron, including a recommended procedure for polishing microsections of cast iron. The report of the subcommittee[5] and the proposed recommended practice[1] are appended hereto. At its meeting in Washington on March 4, Committee A-3 approved the report and the proposed recommended practice and action was taken to refer the recommended practice to Committee E-4 on Metallography for its approval. Committee E-4 approved the recommended practice for submission to letter ballot.

Subcommittee VIII on Cast-Iron Culvert Pipe (W. J. Schlick, chairman) reports that the utilization by the trade of the Standard Specifications for Cast-Iron Culvert Pipe (A 142 - 38) has been investigated and that this standard is being used satisfactorily.

Subcommittee X on Nomenclature and Definitions (A. J. Herzig, chairman) is engaged in the preparation of revisions of the Tentative Definitions of Terms Relating to Cast Iron (A 196 - 36 T). Recommendations for changes will be presented at the June meeting of Committee A-3.

In addition to the terms already covered in Definitions A 196, a study is being made of the advisability of defining other terms relating to testing and mechanical properties as applied to this material.

Subcommitte XI on Methods of Testing (V. A. Crosby, chairman) is reviewing the test methods employed in specifications under the jurisdiction of Committee A-3, having in mind possible correlations of all methods used in these standards. It is expected that besides handling specific questions directly referred to it, Subcommittee XI will become a "clearing house" for review of test methods proposed by other subcommittees of Committee A-3 dealing with both specifications and research.

Subcommittee XV on Impact Testing (J. T. MacKenzie, chairman).—After a study of the drop test method and Charpy impact test method which has been under way for some time, the subcommittee has concluded that both of these methods need to be standardized tentatively for purpose of research and the accumulation of data for possible inclusion in future specifications. The tests proposed are as follows:

Drop Test.—In the drop test the following spans and weights of hammer may be used:

Bar A	4-in. span, $12\frac{1}{2}$-lb. hammer
Bar B	6-in. span, 25-lb. hammer
Bar C	12-in. span, 50-lb. hammer

For the B bar, dimensions of the hammer,[6] anvil, and supports should be approximately those of Fig. 5 in the 1933 report.[7] Each test should be started with the hammer dropping from an initial height of 1 in. and the height of drop should be increased by 1-in. increments, the final drop being reported as the end point.

Charpy Impact Test.—The Charpy impact test should be made as follows: Take half of the transverse bar (1.20 by 21 in.) and cut it into lengths of $8 \pm \frac{1}{8}$ in. Turn or grind the bar to 1.125 ± 0.005 in. round. Place this specimen in the Charpy impact machine observing all the precautions listed in the Tentative Methods of Impact Testing of Metallic Materials (A.S.T.M. Designation: E 23) of the American Society for Testing Materials.[8] The specimens should be broken on 6-in. centers and the results recorded as the energy absorbed by the hammer. Full size bars (1.20-in. round, as cast) may be used as test specimens provided this is explicitly stated in the report.

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[5] See p. 90.

[6] One of the testing machine manufacturers is to be asked for an approximate estimate for the cost of a machine to be built by his company.

[7] Report of Subcommitte XV on Impact Testing of Cast Iron, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 87 (1933).

[8] 1939 Book of A.S.T.M. Standards, Part I, p. 1242.

Subcommittee XVI on Heat Treatment of Cast Iron (J. S. Vanick, chairman) is interested in the work of the recently reorganized Joint Committee on Definitions of Terms Relating to Heat Treatment. The chairman of this subcommittee has been appointed as the representative of Committee A-3 on the Subcommittee on Definitions of Terms Relating to Heat Treatment of Committee E-8 on Nomenclature and Definitions.

Subcommittee XVII on Gray Iron Castings for Valves, Flanges and Pipe Fittings (J. J. Kanter, chairman) is working with Subcommittee VI on correlation of Specifications A 48 with the Standard Specifications for Gray Iron Castings for Valves, Flanges, and Pipe Fittings (A 126 – 40), and a joint report and recommendation will be presented to Committee A-3 at its June meeting.

Subcommittee XVII has made a comparison of the Federal Specifications for Standard Flange Dimensions (Classes 125 and 250 Cast-Iron Flanges; Classes 150 and 250 Bronze Flanges) for Land Use (WW-F-406), Cast-Iron Drainage Pipe Fittings (WW-P-491), Cast Iron (Threaded) Pipe Fittings (WW-P-501a), and 125-lb. Threaded and Flanged Gate Valves for Land Use (WW-V-76b), with the A.S.T.M. Standard Specifications A 126, and has reported that material covered by Specifications A 126 will conform to the material requirements of the above Federal Specifications.

Subcommittee XVIII on Automotive Castings (A. L. Boegehold, chairman) has prepared a revision of the Standard Specifications for Automotive Gray-Iron Castings (A 159 – 40),[4] as mentioned earlier in this report, in order to bring them into closer conformity with the S.A.E. Recommended Practice for Automotive Gray Iron Castings.

Subcommittee XIX on Chilled and White Iron Castings (Johannes Erler, chairman) is continuing its work on the preparation of specifications for chilled and white iron castings.

Subcommittee XX on Soil Pipe (Walker Reynolds, chairman) is working on a revision of the Tentative Specifications for Cast Iron Soil Pipe and Fittings (A 74 – 39 T) and will report at the June meeting of the committee.

Subcommittee XXI on Pressure Pipe (T. H. Wiggin, chairman) is responsible for the Tentative Specifications for Cast Iron Pit-Cast Pipe for Water and Other Liquids (A 44 – 39 T)[2] which are being recommended for adoption as standard as mentioned earlier in this report.

This report has been submitted to letter ballot of the committee which consists of 113 members; 71 members returned their ballots of whom 70 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. BOLTON
Chairman.

J. T. MACKENZIE,
Vice-Chairman.

E. R. YOUNG,
Secretary.

REPORT OF SUBCOMMITTEE VII ON MICROSTRUCTURE OF CAST IRON

PREPARED JOINTLY WITH THE A.F.A. GRAY IRON DIVISION SUBCOMMITTEE ON CLASSIFICATION OF GRAPHITE

The physical metallurgy of gray cast iron is inseparable from considerations of the amount, the size, and the orientation and distribution of graphite in the microstructure of this material. Although all metallurgists familiar with cast iron have long recognized the importance of these factors, no systematic method or recommended practice has previously been accepted generally as a means of determining and standardizing the measurements of the last three. Determination of the chemical content of graphite yields a measurement of the amount of graphite but consideration of this factor alone would mean a failure to recognize the great importance of the microstructure of graphite in determining physical characteristics. Photomicrographs ordinarily can show only a very small area, and inherent variations in the microstructure frequently make representations of graphite by this method either misleading or very tedious and impractical. A more accurate and simpler method seemed very desirable.

Committees of the American Foundrymen's Association and the American Society for Testing Materials have collaborated to prepare this report upon the various aspects of the problem and to develop a recommended practice for evaluating the microstructure of graphite in gray cast iron. The recommended practice includes two series of photomicrographs and drawings covering the complete range in size and type of graphite commonly found in gray irons. A description of a standard procedure to be used in evaluating the microstructure of graphite, and a suggested method for metallographic polishing of gray iron are also included.

The recommended practice does not in any way displace present chemical and physical tests. No discussion is contained on specific effects of microstructure of graphite upon any property of gray cast iron. The work provides only a new testing procedure which should serve as a new tool in aiding investigators in this field.

Historical:

Recognition of the general relationships between graphite flake size, form and distribution on the one hand, and many of the physical properties of cast iron on the other, probably dates back to the very earliest use of the microscope on this material. Much valuable investigative work has been reported in which photomicrographs have been used to illustrate or record the effect of graphite as a controlling variable upon some physical property. References may be found calling attention to the possible usefulness of making actual measurement of the size of graphite flakes and of classifying the various types of structure.

Bolton (**1**)[1] in 1927 described the following general formations: (*a*) broad straight flakes with background of fine flakes, (*b*) rosette or whorl, (*c*) dendritic striation, (*d*) grouped formation, consisting of clear dendritic flakes with fine graphite peninsulas, and (*e*) round temper carbon. He also suggested the following nomenclature for description of graphite formation: (*1*) primary graphite (first deposited formations from liquid phase) retaining characteristic outlines resembling cementitic forms, (*2*) secondary graphite formations, which, although distinctive, have, through influences of diffusion and grain growth, little resemblance to cementitic formations, (*3*) residual graphite (graphite not dissolved in the remelting process) acting as nuclei on resolidification, and (*4*) pseudo-eutectic graphite (finely distributed graphite), probably a decomposition product of ledeburite.

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[1] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this report.

Roll (2) reconstructed the actual volumetric shape of graphite flakes by photographing successive parallel sections separated by known distances. This author proposed the following classification of graphite shapes in cast iron:

1. Isolated flakes, thick and short, scarcely connected.
2. Flakes connected to each other:
   (*a*) Coarse flakes
   (*b*) Network graphite
   (*c*) Rosette-like, globular graphite
   (*d*) Whorl graphite
3. Eutectic graphite:
   (*a*) Network graphite
   (*b*) Globular graphite
4. Temper carbon.

Thum (3) compared graphite flakes with notches as far as their effect upon the physical properties are concerned. He showed six sizes of graphite and gave a stress-strain diagram showing permanent deformation and total deformation for each type. His work seemed to show that the longer the flake, the lower would be the modulus of elasticity. He concluded that both the tensile strength and the modulus of elasticity are determined by graphite content and form.

Norbury and Bolton (4) stated that the terms "fine," "medium," and "coarse" graphite are not sufficiently explicit. The average length of the flakes in a representative area should be stated. They pointed out that it is difficult to measure the length of the flakes very accurately, since the flakes are often curved and join each other, but approximately correct values may be obtained. The flakes may vary considerably in length in different parts of the same casting. The flake length varied from 0.700 to 0.001 mm. in the castings examined.

The British Cast Iron Research Association (4) adopted as far as possible the following magnifications for examination: × 50 diameters, unetched, for showing the general graphite structure. If the graphite is very coarse, × 25, unetched, is occasionally used; if very fine, the specimen is etched and photographed at × 200 or × 1000. A magnification of 200, etched, is used for showing the nature of the matrix. In many irons, if dendritic structure is sufficiently fine, this magnification also may show the general graphite structure. A magnification of 1000, etched, is suggested for revealing the structure of the individual constituents.

Diepschlag (5) differentiated graphite of the following types: (*1*) graphite in large straight plates (hypereutectic graphite) separates while the major portion of the mass is liquid, (*2*) graphite in compact rounded grains separates near the eutectic line if the rate of cooling is slow and the degree of undercooling is slight, (*3*) graphite in thin leaves, with spirals corresponding to the surface of the austenite crystals, and separated in condition of undercooling, (*4*) graphite in a very fine state and equally disseminated (eutectic graphite) separates if the constitution of the alloy is favorable, and (*5*) graphite in soft and porous flakes, often lies adjacent to the cementite, and separates from the solid solution during cooling.

Diepschlag further classified graphite into four groups:

1. Well-defined platy graphite,
2. Graphite in thin plates,
3. Thin curly leaves plus numerous nodulae of graphite, and
4. Eutectic graphite.

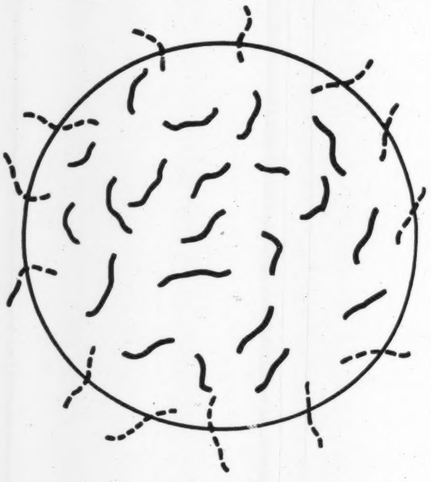
Iitaka and Shiota (6) classified the structure of graphite formed in cast iron as follows: Flake, eutectic, lump, octopus-like, and rosette graphite. They found that the carbon content had a great effect upon the formation of octopus-like graphite. The higher the carbon content, the more frequently it occurred.

At a meeting of the International Committee for Testing Cast Iron held in Paris on June 19, 1937, Prof. A. Portevin presented a paper (7) from which the following is quoted:

> The fundamental importance of the graphite in relation to the strength and other physical properties of cast iron and, consequently the position acquired by micrographic examination in research and foundry control work are well known. Numerous publications concerning the structure of cast iron or calling on micrography have been appearing at conventions and in the journals of foundry associations.
>
> On reading these publications, one is struck by the diversity and, consequently, the vagueness, not to say the incorrectness, of the terms describing the graphite, regardless of the importance of these terms. This results in obscurity, confusion, errors, and sources of sterile discussion.
>
> It is apparent that it would be useful to bring order and method into the manner of defining and characterizing the graphite in cast iron. Such an undertaking falls within the plan of activities of the International Committee for Testing Cast Iron; for its field of study includes all the tests of cast iron, and micrography is, we would say, one of the more important elements of such a study. Therefore, I propose a study in common of this subject.

Portevin then went on to discuss the substance of his course in metallography at the Foundry Technical School, describing a method taught for evaluating the microstructure of graphite. In classifying the important characteristics, he described by means of drawings and methods of measurement, existing variations in (*1*) shape, (*2*) distribution, and, (*3*) size of the graphite particles.

In Fig. 1, one of the several methods that he has discussed for measuring the size of the graphite particles, is described. This method, of course, involves counting individual graphite flakes at a number of positions on a given specimen. Acknowledgment was made to Jeffries who used this method originally in estimations of grain size of metals.



$$N = \frac{N_1 + 0.6N_2}{S} G$$

where:
N = Flake number in flakes per mm².
N_1 = The number of flakes entirely within the circumference.
N_2 = The number of flakes intersected by the circumference.
S = The area enclosed in the circle in mm².
G = The magnification used.

FIG. 1.—A Method for Determining Graphite Flake Number (Portevin).

Portevin included in his classification the temper carbon form of graphite. He made, however, only an indirect reference to orientation of graphite flakes as a variable.

After describing possible mathematical means of arriving at flake size or number by various procedures, Portevin added: "A fineness type scale for graphite could be established by means of photomicrographs expressing fineness as in the case of 'grain size' in steels." In conclusion, he suggested the establishment of:

> 1. Two series of drawings (supplemented where necessary by photomicrographs) showing the forms or aspects characteristic or typical:
>   (*a*) Of the particles of graphite, and
>   (*b*) Of the distribution of the graphite in the cast iron. Each series should carry a designation to be adopted and chosen in accordance with the principles that we have herein recalled.
> 2. A scale of fineness for graphite, supplemented by a method for evaluating the size of the particles.
>
> In this way would be established at the same time exact correspondence between the terms used in different languages from now on.

Portevin's suggestions have since been tried out by Szczawinsky (8) and coworkers who report that the determination of flake size with the aid of the circumference of a circular area is adequate for practical use. These investigators did not favor the method of using a comparison chart of standard photomicrographs.

By coincidence, a few days following the reading of Professor Portevin's paper, a paper by Mahin and Hamilton (9) on the same subject was presented at the 1937 annual meeting of the American Society for Testing Materials in New York City. These authors proposed two standard charts for the evaluation of the graphite variable. One of these consisted of eight photomicrographs taken at a magnification of 100 and showing examples of eight ranges in flake size to be designated as Nos. 1, 2, etc., up to No. 8. The other chart, showing four example photomicrographs, set forth four proposed standard graphite flake types, designating these as types A, B, C, and D.

Both of these charts were based upon experiences with actual sizes and types of graphite observed in a large number of microsections from iron castings embracing a wide range in as-cast section size and alloy type.

This work further emphasized the use of suitable magnifications. It was suggested that a preliminary examination be made of every microsection at low magnification in order to observe the degree of uniformity of flake size or type. A method of recording structure corresponding to that used for mixed grain size in steels was suggested in order to take full cognizance of existing variations. Thus, a microsection showing two areas each having a characteristic flake size and type would be classified by observing the approximate relative areas and the size number and type letter found in each. In the instance illustrated, it was interesting to note how misleading would have been any judgment of the specimen based upon an examination of either of the photomicrographs taken only at a magnification of 100.

In the bibliography in the Appendix, a number of additional references are included. Those most directly associated with the problem, however, have been summarized above.

Committee Action:

At their various joint meetings, the two committees have discussed the question of a standardized classification for graphite in gray cast iron, and have agreed to offer as tentative the Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron which accompanies this report.[2] This is to be sponsored jointly by Committee A-3 of the American Society for Testing Materials and by the American Foundrymen's Association. It includes (*1*) a chart illustrating standard graphite flake lengths, based upon that proposed by Mahin and Hamilton, (*2*) a chart illustrating types of graphite flake structure with respect to distribution and orientation, as proposed by Mahin and Hamilton with one addition suggested by Portevin, (*3*) directions for the use of these charts in practical metallographic work, and (*4*) an appendix describing a suitable method for polishing cast iron specimens so that the character of the graphite flakes can be properly appreciated.

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[2] This recommended practice was accepted as tentative by the Society and appears in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 300.

This work is limited to the flake type of graphite, temper carbon not being considered. The method recommended is that of comparison of a given structure with a set of standards. It was decided not to sponsor the more tedious method of measuring or counting individual graphite flakes in an effort to arrive at a mathematically accurate representation for the field so studied. It was believed that such attempted accuracy is unwarranted in view of inherent nonuniformity in cast iron.

This work does not embrace the possible means of controlling graphite flake type and size and their effects upon mechanical properties. The latter studies will be much more important and far reaching but can proceed effectively only when suitable standards of microstructure are generally available.

The reference standards included in the proposed recommended practice[2] are in no way to be construed as specifications. The preparation of specifications based in any way upon these reference standards must await the time when knowledge is available regarding means of control of graphite type, size, and distribution, and the resultant effects upon mechanical properties. The standards are offered at this time primarily to permit accurate reporting of microstructures of gray iron and to facilitate the comparison of reports by various workers or laboratories.

Acknowledgments:

The recommended practice and charts, is chiefly the work of W. E. Mahin of the Westinghouse Electric and Manufacturing Co., although other members of Subcommittee VII, of Committee A-3, and the A.F.A. Gray Iron Division Subcommittee on Classification of Graphite assisted. The photomicrographs used as illustrations were prepared by J. A. Ferree at the Westinghouse laboratory, and by A. Boyles at Battelle Memorial Institute. During the course of the work, many helpful suggestions were made by J. W. Bolton of the Lunkenheimer Co., Cincinnati, Ohio. Mr. Bolton, assisted by A. J. Smith, prepared the drawings which accompany the photomicrographs. The appendix was written by A. Boyles of Battelle Institute, who developed the method therein described. The historical material and bibliography were prepared by R. R. Adams at Battelle Memorial Institute.

Respectfully submitted on behalf of the subcommittee,

G. F. COMSTOCK,
Chairman.

APPENDIX

BIBLIOGRAPHY ON CLASSIFICATION OF GRAPHITE IN CAST IRON

(1) J. W. Bolton, "Some Graphite Formations in Gray Cast Iron," *Transactions*, Am. Foundrymen's Assn., Vol. 35, pp. 386-409 (1927).

(2) F. Roll, "The Shape of Graphite," *Foundry Trade Journal*, Vol. 41, p. 124 (1929).

(3) A. Thum, "Recent Observations on the Mechanical Properties of Cast Iron," *Die Giesserei*, Vol. 16, pp. 1161-1174 (1929).

(4) A. L. Norbury and L. W. Bolton, "The Measurement of Graphite Size in Cast Iron," *Bulletin No. 26*, British Cast Iron Research Assn., pp. 189-191 (1929) (Bureau Report No. 56).

(5) E. Diepschlag, "The Influence of Graphitization in Cast Iron," *Foundry Trade Journal*, Vol. 41, pp. 23-26 (1929).

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(7) A. Portevin, "Classification of Graphite in Cast Iron," report submitted to International Committee for Testing Cast Iron in Paris, June, 1937.

(8) Szczawinsky, "Application of Portevin's Method of Classifying the Graphite of Cast Iron in Practice with Comments Made by Members of the Test Committee for Castings of the Polish Society for Testing Materials," report submitted to International Foundry Congress at Warsaw, Poland, September, 1938.

(9) W. E. Mahin and J. W. Hamilton, "A Proposed Standard Classification of Graphite in Gray Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Part II, Vol. 37, pp. 52-65 (1937).

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REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel has held two meetings during the past year: one on June 25, 1940, during the annual meeting of the Society in Atlantic City, N. J., and the other on March 4, 1941, at the spring group meetings of A.S.T.M. committees in Washington, D. C.

Since the last report, 2 new members have been elected and 1 member has resigned; the committee membership now totals 128, of whom 58 are classified as producers, 42 as consumers, and 28 as general interest members.

I. Adoption of Tentative Standards as Standard

The committee recommends that the following three tentative standards be approved for reference to letter ballot of the Society for adoption as standard, with revisions in the two specifications as indicated below:

Tentative Specifications for Zinc-Coated Steel Wire Strand ("Galvanized" and "Extra Galvanized") (*A 122 – 39 T*),[1] revised as follows as proposed by Subcommittee VI to supersede the present Standard Specifications for Zinc-Coated (Galvanized) Iron or Steel Wire Strand (Cable) (A 122 – 33):

Title.—Change to read as follows:

> Specifications for Zinc-Coated Steel Wire Strand ("Galvanized" and Class A ("Extra Galvanized")).

Section 16.—Omit Paragraph (*a*) which reads as follows, renumbering Paragraph (*b*) as Section 16:

> (*a*) The permissible variations from the calculated diameter of the strand shall conform to the requirements prescribed in Table IV.

Table IV.—Omit Table IV which lists the permissible variations in diameter of finished strand and renumber Table V accordingly, changing the reference to Table V in Section 16 (*b*) to Table IV.

Section 18.—Change the last sentence to read as follows by the addition of the italicized words:

> Each coil or reel shall have a strong tag securely fastened to it showing the length, size, grade of the strand, class of coating, *A.S.T.M. designation A 122*, and the name or mark of the manufacturer.

Tentative Specifications for Zinc-Coated Steel Wire Strand (Class B and Class C Coatings) (*A 218 – 39 T*),[2] revised as follows as proposed by Subcommittee VI:

Section 16 and Table IV.—Make the same changes as recommended above for Specifications A 122.

Section 18.—Change the last sentence to read as follows by the addition of the italicized words:

> Each coil or reel shall have a strong tag securely fastened to it showing the length, size, grade of the strand, class of coating, *A.S.T.M. designation A 218*, and the name or mark of the manufacturer.

Tentative Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (*A 239 – 40 T*),[3] proposed by Subcommittee VII to supersede the Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Wire (A 191 – 38), and the Tentative Method of Test for Uniformity of Coating by the Copper Sulfate Dip Test (Preece Test) on Zinc-Coated (Hot-Galvanized) Steel Castings and Forgings, Gray-Iron and Malleable-Iron Castings (A 208 – 38 T).

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 917.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 922.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 288.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 128 members; 94 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. ADOPTION OF TENTATIVE STANDARDS AS STANDARDS			
Spec. for Zinc-Coated Steel Wire Strand ("Galvanized" and "Extra Galvanized") (A 122 – 39 T), as revised, to supersede Standard Specifications A 122 – 33	71	1	22
Spec. for Zinc-Coated Steel Wire Strand (Class B and Class C Coatings) (A 218 – 39 T), as revised	67	3	24
Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles (A 239 – 40 T), to supersede Standard Method A 191 – 38 and Tentative Method A 208 – 38 T	76	0	18

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Inspection of Annapolis Tests (E. S. Taylerson, chairman).—The specimens of copper-bearing and non-copper-bearing corrugated black sheets that have been exposed at Annapolis, Md., since October 17, 1916, were inspected on May 6, and on October 14, 1940. At the May inspection it was found that 12 sheets had failed: namely, C16, D1, H2, H9, H16, M11, M12, M13, T2, T5, T13 and Y2. In October 17 sheets were recorded as failures: namely, B28, E4, H3, H11, I2, M1, M3, M4, M5, M7, M8, M9, T8, T9, T11, T12, and V5. All of the failures to date are in the No. 22-gage material. A summary of the failures to date is given in the accompanying Table II.

Subcommittee V on Total Immersion Tests (W. J. Jeffries, chairman).—With the exception of sample plates which were built into tank ships, all of the specimens exposed in the various tests carried on by this subcommittee have now been removed and the tests discontinued.

A final report on the behavior of the low-copper and high-copper seamless steel pipe specimens which were exposed from 1927 to 1939 in sea water at Portsmouth, N. H., and Key West, Fla., has been completed and is being given further study. It will be ready for publication next year.

A final report is still to be made on the riveted plate test specimens and on the plates built into tank ships. The subcommittee plans to submit these reports and a report summarizing the results obtained from all of the tests carried out by the subcommittee for the 1942 annual report.

Subcommittee VI on Specifications for Metallic-Coated Products (T. R. Galloway, chairman).—As is indicated earlier in this report, the subcommittee proposes that Specifications A 122 – 39 T and A 218 – 39 T covering galvanized strand be adopted as standard with minor changes, and that Standard Specifications A 122 – 33 be discontinued.

The Section on Zinc-Coated Sheets is actively engaged in an attempt to reconcile differences between the A.S.T.M. and Federal specifications and hopes to propose a revision of the Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 38 T) next year. The specifications will,

TABLE II.—RECORD OF FAILURES IN ATMOSPHERIC EXPOSURE TESTS AT ANNAPOLIS OF NO. 22-GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed October 17, 1916. Inspections Made Semiannually from 1917 to 1941.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test[a]	Life of Each Failed Sheet, yr.																Number of Sheets in Test[a]	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z......	Open-hearth steel	B	0.032	0.32	0.063	0.042	..	0.019	3	13.5	17.0	19.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	16.5	......Z
O.....	Open-hearth steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	..	..	..	..	12	12	10.4	......O
B.....	Open-hearth steel	A	0.09	0.38	0.017	0.062	0.036	0.084	3	20.0	20.5	24.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	21.5	......B
M.....	Open-hearth steel	B	0.093	0.419	0.013	0.041	0.011	0.185	13	20.0	20.5	23.5	23.5	23.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	*	..	..	..	13	12	..	......M
B.....	Open-hearth steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	19.5	20.0	21.5	22.0	22.5	23.0	*	*	*	..	..	..	..	..	..	..	9	6	..	......B
Z.....	Open-hearth steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
T.....	Open-hearth steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	19.5	19.5	19.5	21.5	21.5	21.5	23.0	23.5	23.5	23.5	24.0	24.0	24.0	24.0	..	..	14	14	22.3	......T
K.....	Open-hearth steel (acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	14	*	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	14	0	..	......K
H.....	Open-hearth steel	A	0.069	0.387	0.016	0.027	0.004	0.244	10	19.5	21.0	21.5	23.0	23.5	23.5	23.5	24.0	24.0	*	..	..	..	..	..	..	10	9	..	......H
Z......	Open-hearth steel	B	0.065	0.35	0.050	0.059	0.003	0.264	2	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	0	..	......Z
Z......	Open-hearth steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
S......	Open-hearth iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	..	..	14	14	10.6	......S
D.....	Open-hearth iron	A	0.02	0.026	0.004	0.022	0.002	0.024	6	16.0	16.0	16.0	19.5	19.5	23.5	..	..	..	..	..	..	..	..	..	..	6	6	18.4	......D
X.....	Open-hearth iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	..	..	14	14	15.7	X
C.....	Open-hearth iron	A	0.01	0.028	0.002	0.027	0.002	0.141	3	17.5	20.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.5	C
C.. ..	Open-hearth iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	16.5	16.5	18.5	19.0	19.0	20.0	20.5	21.5	21.5	..	..	..	..	..	..	..	9	9	19.2	C
U.....	Open-hearth iron	B	0.017	0.074	0.008	0.037	0.005	0.260	13	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	21.0	21.0	22.5	..	..	..	13	13	19.3	......U
V.....	Open-hearth iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	20.5	21.0	22.0	22.0	22.5	24.0	..	..	14	14	20.0	V
A.....	Bessemer steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	..	15	15	18.2	......A
Z......	Bessemer steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	11.5	11.5	11.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	11.5	......Z
Z......	Bessemer steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
I......	Bessemer steel	A	0.041	0.365	0.097	0.068	0.008	0.252	15	24.0	*	*	*	*	*	*	*	*	*	*	*	*	*	*	..	15	1	..	I
Z......	Bessemer steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
Z......	Bessemer steel	B	0.055	0.38	0.110	0.046	..	0.527	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	......Z
Y.....	Wrought iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	18.0	19.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.3	......Y
E.....	Wrought iron	A	0.033	0.034	0.114	0.021	0.134	0.283	10	19.5	20.5	21.0	21.0	23.0	23.0	23.0	23.0	24.0	*	..	..	..	..	..	..	10	9	..	......E
							Total.......		214																Total..	214	163		

* Indicates unfailed sheets.

[a] The number of sheets in the test indicated in this table is less than the number shown in earlier reports because certain sheets were discarded due to mechanical damage.

therefore, be continued as tentative for another year.

Proposed revisions in the Standard Specifications for Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized After Weaving (A 117 - 33) are developing slowly. The basis for specifications for chain-link fence galvanized after weaving has been discussed, but a satisfactory conclusion has not yet been reached.

Discussion is under way of proposed changes in the sampling methods incorporated in the Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 - 40 T), and for Electrodeposited Coatings of Cadmium on Steel (A 165 - 40 T). These specifications will, therefore, be continued as tentative for another year.

The Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 40 T) have been transferred to the jurisdiction of the new Committee B-8 on Electrodeposited Metallic Coatings.

Mr. B. J. Barmack has been appointed chairman of a Section on Galvanized Hardware, and it is expected that the organization of this section will be completed shortly.

Subcommittee VII on Methods of Testing (R. W. Baker, chairman).—As indicated earlier in this report, this subcommittee is proposing that the Preece test Method A 239 - 40 T be adopted as standard and that Method A 191 - 38 covering the Preece test for wire products and Method A 208 - 38 T covering the Preece test for castings, forgings, etc., be withdrawn.

Jurisdiction over the Tentative Methods of Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 - 40 T) has been turned over to the newly organized Committee B-8 on Electrodeposited Metallic Coatings.

Subcommittee VIII on Field Tests of Metallic Coatings (R. F. Passano, chairman).—The major part of the subcommittee's report for this year is concerned with the observations of the Wire Inspection Committee. Its report is appended hereto. From observations made so far on specimens removed from the wire tests (25 per cent of the specimens have been removed from Pittsburgh, Pa., and 10 per cent from Bridgeport, Conn.), the following tentative conclusions seem justified:

1. On zinc-coated specimens the amount of protection increases with increase in the weight of coating.
2. The rate of loss of coating on zinc-coated specimens is greater on small diameter wires than on the larger diameter wires.
3. The rate of loss of base metal is more rapid on the smaller diameter wires than on the larger diameter wires.
4. The relative rate of corrosion at Pittsburgh, Pa., is approximately six times that at State College, Pa., while Bridgeport, Conn., and Sandy Hook, N. J., are about twice as active as State College.

Inspection of the hardware specimens indicates that many of these specimens have served their useful life. They will, therefore, be removed from the test. Those of the remaining specimens in which there is a continuing interest will be retained, but the specimens will be consolidated into a smaller place. It is expected that a report on the hardware test will be included in the next annual report of the committee.

Inspections of the sheet specimens have been carried out. Special attention has been given to the unusual type of failure from underneath the sheet which was observed at Key West, Fla., and reported last year[4] and to the loss-of-weight determinations at Sandy Hook,

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 106 (1940).

N. J., in the spring and in the fall to determine whether there is a difference in rate of corrosion during the summer and winter seasons.

The subcommittee has been doing some work on analyses of the coatings on sheet specimens which were retained when the original specimens were exposed to the atmosphere. Unexplainable differences in the analyses have occurred in the results given by spectroscopic methods and wet chemical methods. A section of the subcommittee has, therefore, been appointed to consult with the National Bureau of Standards which made the referee analyses for the wire test specimens.

At the request of Subcommittee VI, some hand-dipped specimens of zinc-coated sheets are being prepared with varying amounts of aluminum in the coating. It is planned to expose these to see whether any difference in behavior can be traced to the amount of aluminum in the coating.

This report has been submitted to letter ballot of the committee which consists of 128 members; 94 members returned their ballots, of whom 88 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. FINKELDEY,
Chairman.

T. R. GALLOWAY,
Secretary.

REPORT OF WIRE INSPECTION COMMITTEE ON FIELD TESTS OF WIRE AND WIRE PRODUCTS

(Covers 1939 and 1940 Inspections)

NOTE.—Reference to the previously published report of Subcommittee VIII on Field Tests of Metallic Coatings, of Committee A-5 on Corrosion of Iron and Steel, which appeared in the 1939 *Proceedings*,[1] will be helpful in certain studies of this exposure test because it contains all the data assembled in inspections prior to 1939, descriptions of the test methods as well as photomicrographs and other characterizations of specimens in the test.

This report presents the results of the atmospheric corrosion tests on wire and wire product specimens after exposure for about four years at each of eleven locations. These eleven test sites have atmospheres of divers types, including severely industrial, mildly industrial, seacoast, and rural. At each location more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations; farm-field fence at nine sites; barbed wire at eight locations; and chain-link fence at eight places. Previous reports[2] show the details of the locations and of the number and nature of the test specimens involved.

The extent of corrosion on specimens is being measured by the following:

1. Visual examination at the test locations either semiannually or annually, depending on the corrosiveness of the test site atmosphere. At the time of inspection, records are made of the appearance of every wire and wire product specimen at each test site. The records are

(*a*) Qualitative descriptions prior to the time of the appearance of rust or rust-resembling corrosion products, that is, the specimens are noted as appearing "metallic," "gray," "brown," "black," etc.

(*b*) Quantitative estimations after the appearance of rust or rust-like corrosion products. Estimates are made of the percentage of area in each specimen that has actually rusted and the percentage that is "yellowed," seemingly by iron corrosion products, but has not yet shown actual rusting of the base metal.

2. Tension tests on the unfabricated wire specimens. These are the specimens exposed in heptads, that is, seven specimens taken from the same lot of wire and supposedly alike. An attempt is made to remove and test the first specimen of each heptad when it has lost 5 to 10 per cent of its strength; the seventh specimen, when it has lost some 75 per cent of its strength; and the intermediate five specimens when they have lost suitable proportions of their strength.

The records of progressive loss of strength of the unfabricated wire specimens will afford an indication of the rate of loss of strength of the wires in the farm fence and barbed wire specimens, because wire from nearly every lot used in the fabrication of the fences and barbed wires is also under test as unfabricated wire.

3. Loss of weight tests are being made on lengths of zinc-coated wire (each about 42 in. long) carefully weighed and measured before exposure. There are 144 wires (16 alike of each of 9 wire lots)

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 99 (1939).

[2] *Ibid.*, p. 156.

exposed at each of the locations of Pittsburgh, Pa., Sandy Hook, N. J., and State College, Pa. These wires originally carried weights of coating of 0.66 to 1.04 oz. per sq. ft. The working scheme for handling these specimens at each location is to remove one-fourth at a time—the first quarter when they have lost a relatively small percentage of their coating; the last quarter when most of the coating has gone but before significant rusting has occurred; and the intermediate two one-quarter lots when they have lost suitable proportions of their coating.

Semiannual inspections have been made at Pittsburgh, Pa., Bridgeport, Conn., Sandy Hook, N. J., and State College, Pa., by a traveling committee and annual inspections have been made at the other test sites by the university people in charge of the test plots. The roster of these university people remains the same as that published in the 1939 report,[1] except that L. W. Neubauer has replaced J. D. Long as the Wire Inspection Committee's representative in charge of the University of California test sites at Davis and Santa Cruz, Calif.

Findings from Visual Examination for Corrosion at the Test Plots:

The detailed corrosion inspection records collected in 1939 and 1940 are presented in Table I for unfabricated wire specimens, Table II for barbed wire, Table III for farm-field fence, Table IV for chain-link fence, and Table V for wire strand. A study of this mass of detailed data brings out certain significant observations and inferences noted below.

Peculiarities of the Data.—Occasionally a specimen which has become yellowed or rust stained has been reported as rusted on a particular inspection date and then judged to be not yet actually rusted at the time of the next inspection six months later. Table I identifies all such specimens (a total of 17) by an asterisk. A review of the data indicates that the premature observations of rust were made largely by a few members of the inspection committee who were less experienced than others who have been inspecting zinc-coated sheets, hardware, or wire products for several years.

These occasional reports of premature rusting should have no bearing on final interpretations of data or conclusions that may be drawn. They become potential causes of misinterpretation only when the data of individual reports are considered without reference to the data of later reports.

Unfabricated Wire Specimens Carrying Zinc Coatings.—At Pittsburgh, complete rusting has occurred on all specimens carrying coatings of 1.0 oz. per sq. ft. or less; partial rusting has taken place on specimens in the group range of 1.2 to 1.8 oz. per sq. ft. coatings; but no rusting has started on specimens carrying coatings of 2 oz. per sq. ft. or more. At Bridgeport and at Sandy Hook, complete or nearly complete rusting has occurred on specimens in the groups carrying coatings of 0.45 oz. per sq. ft. or less; partial rusting has taken place on specimens in the groups having coating in the range of 0.45 to 0.60 oz. per sq. ft. at State College, Pa., and at Lafayette, Ind., rusting is beginning on a few of the lightest coated specimens (0.20 to 0.35 oz. per sq. ft. range). At all of the other test sites all zinc-coated unfabricated wire specimens are still unrusted.

Table A summarizes the corrosion condition of the zinc-coated unfabricated wire specimens after exposure for about four years at each of the eleven test sites.

Figure 1 shows the progressive development of rust of all groups of wires which have become 100 per cent rusted. In this figure there are two instances in

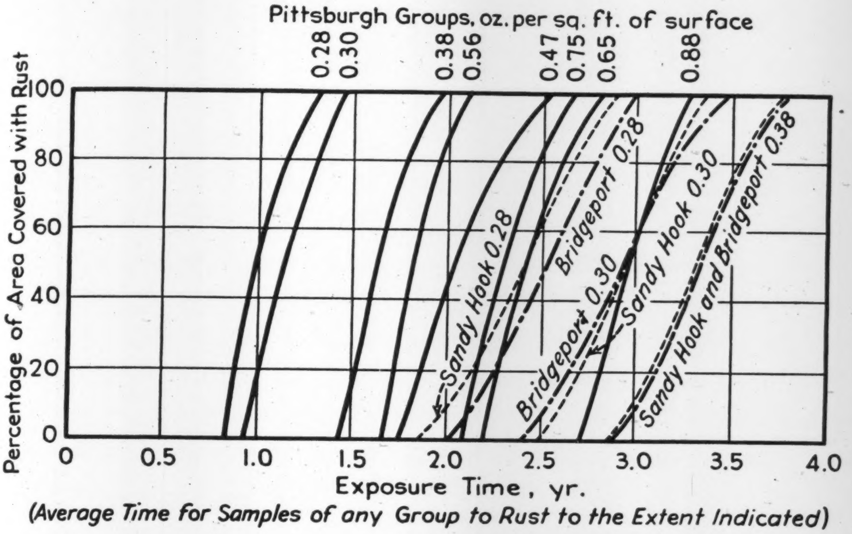

FIG. 1.—Progressive Development of Rust on Zinc-Coated Unfabricated Wire.
Coating weight groups in which all of the samples are rusted.

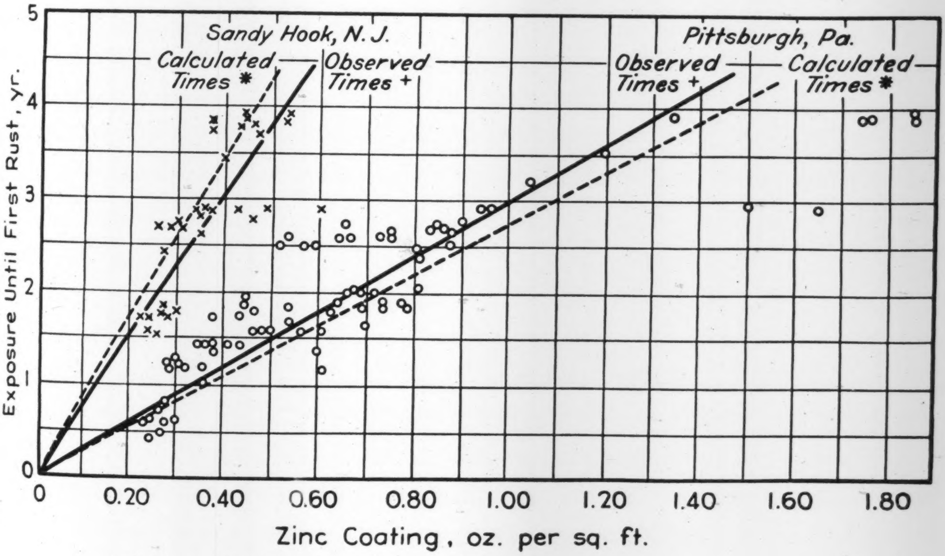

FIG. 2.—Exposure Time for First Rust to Occur.
All unfabricated wire lots exposed at Pittsburgh and at Sandy Hook that show rust.

* Time (years) when first rust should appear, calculated from data of weight loss tests. (Samples at Pittsburgh lose 0.369 oz. per sq. ft. per year; at Sandy Hook, 0.117 oz. per sq. ft. per year.)

† Straight line curve drawn through origin and average point of all observations.

which a lighter-weight coating group outlasted a heavier-weight group—the 0.65-oz. Pittsburgh group lasted longer than the 0.75-oz. group, and the 0.47-oz. group longer than the 0.56-oz. group. In both instances, the lighter-weight, longer-lasting group is made up of heavier gage wires (Nos. 6, 9, and 11 gage) and the shorter lasting group of lighter gage wires (Nos. 12½ and 14½ gage). These reversals are not inconsistent with other findings brought out in this report.

TABLE A.—CORROSION CONDITIONS OF ZINC-COATED UNFABRICATED WIRES AFTER APPROXIMATELY FOUR YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states.

Exposure Sites	Coating Group, oz. per sq. ft. of surface										
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 to 0.85	0.80 to 1.00	1.20 to 1.35	1.60 to 1.80	2.00 or more
Pittsburgh, Pa.	100R	100R	100R	100R	100R	100R	100R	100R	48R	4R	G
Sandy Hook, N. J.	100R	92R	100R	21R	13R	GY	GY	GY	G	G	G
Bridgeport, Conn.	100R	96R	100R	14R	11R	GY	GY	GY	G	G	G
State College, Pa.	GY	5R	GY	GY	G	G	G	G	MG	MG	MG
Lafayette, Ind.	2R	2R	G	G	G	G	MG	MG	MG	MG	MG
Ames, Iowa	G	G	G	G	G	G	G	MG	G	MG	MG
Manhattan, Kans.	G	G	G	G	MG	G	MG	MG	MG	MG	MG
Ithaca, N. Y.	G	G	G	G	MG	G	MG	MG	M	M	M
Santa Cruz, Calif.	G	G	G	G	MG	MG	MG	M	....	M	M
College Station, Tex.	MG	MG	MG	MG	MG	G	MG	G	G	MG	G
Davis, Calif.	G	G	G	G	MG	MG	MG	MG	....	M	M

Figure 2 shows the time for appearance of first rust in relation to the weights of coating on all specimens at Pittsburgh and at Sandy Hook which have started to rust. Each point on the plotting represents an individual specimen. If the time required for rust to appear was always proportional to the coating weight, then all of the points would lie on the straight line (unbroken line) drawn from the origin through the average point of all of the points.

Unfabricated Wire Specimens Carrying Coatings Other Than Zinc.—The copper-covered wires show no rusting at any location. The uncoated corrosion-resistant steel wires show superficial corrosion or yellowing (often in a spotted pattern) at several locations. Generally the 18 per cent chromium, 8 per cent nickel alloy steel wires retain their original metallic luster longer than the 13 to 14 per cent chromium alloy steel wires. Nearly all of the lead-coated wires at all locations show some pin-hole rusting.

Barbed Wire and Farm-Field Fence.—These products are exposed at all locations except Pittsburgh, Sandy Hook, and Bridgeport. The specimens in the 0.20 to 0.30 oz. per sq. ft. group show some rust at Lafayette, and rusting on the barbs has occurred among specimens of this group at State College, Ithaca, and Santa Cruz. The lead-coated barbed wire specimens show some pin-hole rust at most locations.

Rusting of farm-field fence specimens has started among the two lightest weight coat groups (0.20 to 0.30 and 0.25 to 0.35 oz. per sq. ft.) at Lafayette. Also, fence No. 215 shows slight rusting at State College, Ithaca, and Davis. Lead-coated fences generally show the pin-hole rusting and yellowing characteristic of all lead-coated specimens in the test.

The corrosion performance of any individual specimen of barbed wire or of farm-field fence listed in Tables II and III may be directly compared with the performance of specific unfabricated wire specimens. The interior line and stay wires of every fence, the top and bottom line wires of every fence, and the line wires of every barbed wire specimen are made of wire of an identified lot number. These same lots of wire are usually under test as unfabricated wire. The Reference Information Table (Plate I) in this report shows the numbers of the wire lots used in fabricating all fence and barbed wire samples.

Chain-Link Fence.—Chain-link fence is exposed at all locations except Ames, Manhattan, and College Station. At Pittsburgh rust is present on all the zinc-coated specimens except two carrying heavy coatings. The corrosion-resistant steel specimen is unrusted, and no rust had appeared on the copper-covered specimen up until the time of its unauthorized removal in 1939. At Sandy Hook and at Bridgeport only the three lightest zinc-coated specimens and the lead-coated specimens show any rust. At the other locations only the lead-coated specimens show any rust.

Wire Strand.—Wire strand, exposed at all locations, shows pin-hole rusting on the lead-coated strand at all locations and rust on the four lightest zinc-coated specimens only at Pittsburgh.

TABLE B.—LOSS OF ZINC COATING, OZ. PER SQ. FT. PER YR. OF EXPOSURE.[a]

Wire Lot	Original Coating Weight,[b] oz. per sq. ft.	Pittsburgh, Pa.[c]				Sandy Hook, N. J.			State College, Pa.	
		1.48 yr.	1.94 yr.	2.45 yr.	2.93 yr.	1.44 yr.	2.41 yr.	3.45 yr.	1.30 yr.	3.31 yr.
				GROUP A[a]						
No. 148	0.88 to 0.97	0.343	0.354	0.314	.....	0.114	0.107	0.104	0.069	0.058
No. 346	0.84 to 0.96	0.398	0.392	0.364	.....	0.119	0.113	0.112	0.062	0.059
No. 542	0.68 to 0.71	0.448	.....	.....	.....	0.136	0.114	0.118	0.077	0.059
				GROUP B						
No. 247	0.91 to 1.01	0.349	0.355	0.337	.....	0.118	0.126	0.126	0.084	0.063
No. 345	0.76 to 0.97	0.376	0.365	0.329	.....	0.137	0.131	0.127	0.084	0.062
No. 541	0.68 to 0.81	0.395	0.374	0.326	.....	0.142	0.139	0.134	0.076	0.058
				GROUP C						
No. 248	0.66 to 0.92	0.349	0.315	.....	.....	0.107	0.095	0.099	0.051	0.050
No. 348	1.02 to 1.07	0.359	0.343	0.368	0.328	0.112	0.099	0.100	0.065	0.049
No. 540	0.68 to 0.72	0.428	0.377	.....	.....	0.133	0.105	0.106	0.074	0.046

[a] The wires listed under groups A, B, or C are closely alike as regards base metal composition and coating characteristics.

[b] Zinc coating weight determinations as given in the Report on Original Characteristics of Wire and Wire Products Involved in Field Exposure Tests, see *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 101 (1939).

[c] Blank spaces under Pittsburgh heading indicate no data available because rusting had started at the times of removal dates.

Findings from Weight Loss Specimens:

At the start of the test 144 wires (16 alike of each of 9 wire lots) were exposed at Pittsburgh, at Sandy Hook, and at State College. The test procedure plan was to remove the specimens one-fourth at a time from each location, taking the last quarter when the coating was nearly gone but before rusting started. All of the removals have been made from Pittsburgh (unfortunately rusting had begun on some specimens before the third and fourth removals were made). Table VII shows the results obtained from the third and fourth removals from Pittsburgh, from the second and third removals from Sandy Hook, and from the second removals from State College. The earlier removals from these three locations were reported on in the 1939 report.[2]

Reference to summary Table B shows the following:

1. There is a tendency for the lighter gages of wire to lose coating somewhat faster than the heavier gages.

2. There is an indication that there is

slight decrease in the rate of coating loss with increasing periods of exposure.

3. The over-all average annual loss of coating in ounces per square foot of surface at Pittsburgh is 0.369; at Sandy Hook it is 0.117; and at State College it is 0.063.

4. There is not a consistent indication that any one of the three groups (A, B, or C) loses coating at a more rapid rate than the other two, although the data might be interpreted to indicate that group C loses weight at the lowest rate.

5. The time required for first rust to appear on specimens thus far rusted at Pittsburgh and at Sandy Hook checks reasonably well with the time that would be expected from the data of the weight loss specimens. The observed times at Pittsburgh are about 9 per cent longer than the calculated times and at Sandy Hook are about 11 per cent shorter than those calculated, as is shown in Fig. 2.

Findings from Specimens Removed for Tension Tests:

There were originally 840 tension strength specimens exposed at each site, except Lafayette and College Station (833) and Davis and Santa Cruz (798). In the 1939 report[2] there were reported the results of tension and elongation tests on 388 wires removed prior to 1939. Since then 345 additional wires have been removed, divided among the different test sites as shown in Table C. The detailed results of the tests on these 345 wires are shown in Table VI.

Pittsburgh is the most corrosive of the test sites, with Sandy Hook rating next, and Bridgeport next. Of the eight test stations at colleges, Lafayette and State College appear the most corrosive. The remaining six college stations are all mildly corrosive but have not yet yielded sufficient data to definitely establish an order of rating. Table D intercompares the test sites as regards percentage of loss of tensile strength per year on bare wires of different gages. The figures in this table should not be used for predicting the number of years wires will last at any given site because there is usually a tendency for the rate of strength loss to decrease as the years of exposure increase.

The studies to date have not developed an explanation of the findings reported in 1939 that many of the specimens increased slightly in strength after the original strength tests were made on the specimens. This strength increase tendency has now substantially completely stopped. However, a few specimens recently removed from the least corrosive sites still show a slightly higher strength than their companion specimens tested before exposure (maximum increase, 4 per cent).

TABLE C.—NUMBER OF TENSION SPECIMENS REMOVED FOR TEST.

Exposure Site	Number of Wires Removed 1939 to 1940				Removed During 1937 to 1939	Total Removed to Date
	Bare Steel	Lead Coated	Zinc Coated	Total 1939 to 1940		
Pittsburgh, Pa.	47	4	40	91	120	211
Sandy Hook, N. J.	41	4	8	53	56	109
Bridgeport, Conn.	26	4	7	37	49	86
State College, Pa.	19	0	0	19	26	45
Lafayette, Ind.	30	1	0	31	19	50
All Other Locations[a]	114	0	0	114	118	232
Total	277	13	55	345	388	733

[a] From each of the six remaining sites (Ithaca, Ames, Manhattan, College Station, Davis, and Santa Cruz) 19 bare steel wires were removed, total 114 wires.

Tension tests on 32 lead-coated wires removed to date show no significant loss of strength for any of these wires.

In Table E is arranged a comparison of certain classes of specimens, as regards strength loss and calculated annual diameter decreases, the classes involved being those exposed at Pittsburgh, Sandy Hook, and Bridgeport from which three or more successive removals have been made.

The first removals of all uncoated wires from these sites were made at the end of about one and one-half years. During this period, all had lost a much larger proportion of their original strength than they did in any subsequent corresponding period. (In Table E, in any of the columns headed with A's or B's compare the strength loss figures for 1.48 and 2.93 yr.)

TABLE D.—LOSS OF TENSILE STRENGTH SHOWN BY EXPOSED UNCOATED STEEL WIRE.
Loss in Tensile Strength Expressed in per cent per year of exposure (approximately four years exposure).

Wire Lot	Original Breaking Load, lb.	Pittsburgh, Pa.	Sandy Hook, N. J.	Bridgeport, Conn.	State College, Pa.	Lafayette, Ind.	Ames, Iowa	Ithaca, N. Y.	Manhattan, Kans.	College Station, Tex.	Santa Cruz, Calif.	Davis, Calif.
COPPER-BEARING BARE STEEL												
No. 506	520	13.9	10.6	7.2	7.0	5.7	3.8	3.2	1.5	2.3	2.1	0.7
No. 507	595	15.2	12.2	7.8	5.8	5.6	3.1	2.0	0.2	2.4	2.7	0.2
No. 405	485	9.5	7.7	4.6	3.2	3.8	2.3	1.3	0.0	0.5	0.3	0.6
No. 406	470	8.2	6.5	3.7	2.3	2.4	1.3	2.1	0.0	0.8	0.3	0.0
No. 305	1085	7.0	4.8	3.9	3.0	2.6	1.5	1.7	0.1	1.4	1.0	0.3
No. 306	710	5.7	4.3	2.0	1.2	1.9	1.4	1.2	0.0	0.0	0.2	0.0
No. 205	1110	5.8	4.3	3.9	1.0	1.9	0.2	2.3	0.5	0.7	0.5	0.0
No. 206	1480	5.1	4.3	3.9	2.9	3.1	1.8	2.4	0.8	0.6	0.3	0.6
No. 102	2095	5.3	3.8	3.0	2.9	2.8	2.1	1.9	1.5	1.8	1.3	0.7
No. 106	2400	2.8	3.1	1.0	1.0	1.0	0.0	0.0	0.0	0.3	0.7	0.0
LOW-COPPER CONTENT BARE STEEL												
No. 501	500	15.8	10.4	8.2	5.8	6.2	3.3	2.7	1.3	1.6	1.8	0.3
No. 400	480	9.3	6.7	3.5	1.8	2.6	1.5	1.3	0.0	0.5	0.3	0.6
No. 401	450	11.8	7.7	4.5	1.5	3.0	1.9	1.4	0.0	0.9	0.9	0.0
No. 300	740	6.8	5.2	2.8	2.1	1.8	1.6	1.4	0.0	0.5	4.0	0.0
No. 302	1100	9.1	6.3	5.2	5.6	4.0	2.8	3.1	1.0	2.3	1.9	1.8
No. 200	1045	7.7	4.6	5.5	2.9	3.5	2.0	2.7	0.9	0.5	0.9	0.5
No. 202	1640	6.7	2.8	4.4	1.4	1.7	1.1	1.9	0.2	0.8	0.0	1.0
No. 101	2315	5.2	3.4	2.7	1.2	1.7	0.9	0.9	0.1	1.1	0.0	0.0
No. 105	2760	4.7	3.5	2.9	1.7	2.6	0.7	0.7	1.3	1.4	0.9	0.6

The first removals of zinc-coated specimens were usually made soon after rusting started. In any unit length of time after rust appeared, the specimens had lost a larger proportion of their strength than they did in any subsequent corresponding period of time. (Intercompare the figures in column C of Table E, using the time figures in column T_r to define what figures in column C should be intercompared.)

The copper-bearing uncoated wires at Pittsburgh are continuing to corrode less rapidly than the low copper content specimens, but the difference does not yet appear significant at the other locations. Even at Pittsburgh the difference is not very large. (In Table E, compare the horizontal figures in columns A_1 and B_1, or A_2 and B_2, or A_3 and B_3.)

The 1939 report[2] stated that (at that time) there was "no very clear-cut indication that the lighter gage uncoated wires were being penetrated more rapidly than the heavier gage ones." By now there appears to be a trend for the lighter gage wires to undergo the more rapid penetration. (In Table E, note the figures within any column headed by D, E's, or F's, comparing the figures for the different gages at corresponding exposure times.)

The penetration experienced by zinc-coated wires in a unit time after rust starts appears to be usually about the same as or slightly less than the penetration undergone by bare wires in a unit time after exposure. Just how precise

TABLE E.—TABULATION ARRANGED TO COMPARE CLASSES OF SPECIMENS AS REGARDS CUMULATIVE STRENGTH LOSSES AND CALCULATED ANNUAL DIAMETER DECREASES.

Gage of Wire	Exposure of Uncoated Wires, yr.	Cumulative Strength Losses after Exposure for Periods Indicated, per cent							T_r	Calculated Diameter Decreases, in. per yr.[c]						
		Bridgeport, Conn.		Sandy Hook, N. J.		Pittsburgh, Pa.			Time Since Rust Started on Zinc-Coated Specimens, yr.	Pittsburgh, Pa.			Sandy Hook, N. J.		Bridgeport, Conn.	
		A_1 Low Copper	B_1 Copper Bearing	A_2 Low Copper	B_2 Copper Bearing	A_3 Low Copper	B_3 Copper Bearing	C Zinc Coated		D Zinc Coated	E_1 Copper Bearing	F_1 Low Copper	E_2 Copper Bearing	F_2 Low Copper	E_3 Copper Bearing	F_3 Low Copper
No. 14½	1.48[a]	16.0	19.4	22.0	23.8	36.0	32.0	17.4	(0.71)[b]	0.0080	0.0087	0.0096	0.0065	0.0060	0.0050	0.0042
	1.94[a]	23.0	17.5	24.0	25.5	39.9	34.0	21.9	(1.17)	0.0074	0.0074	0.0080	0.0052	0.0049	0.0035	0.0047
	2.45[a]	21.0	21.0	25.0	29.5	46.0	38.0	29.1	(1.68)	0.0066	0.0061	0.0075	0.0048	0.0040	0.0034	0.0034
	2.93[a]	24.0	...	32.0	34.5	49.0	46.0	35.0	(2.16)	0.0063	0.0062	0.0065	0.0047	0.0043	...	0.0032
	3.48[a]	...	26.0	36.0	39.5	55.0	51.0	39.8	(2.71)	0.0057	0.0058	0.0063	0.0044	0.0041	0.0029	...
No. 12½	1.48[a]	16.0	16.2	18.2	19.3	27.0	22.5	10.0	(0.56)	0.0089	0.0074	0.0090	0.0066	0.0062	0.0053	0.0055
	1.94[a]	10.7	11.0	15.0	15.2	24.2	22.5	12.3	(1.02)	0.0060	0.0056	0.0060	0.0038	0.0038	0.0027	0.0026
	2.45[a]	...	...	16.1	15.2	28.2	23.0	...	...	...	0.0047	0.0058	0.0031	0.0034	...	...
	2.93[a]	...	12.0	...	...	35.1	30.3	20.6	(2.01)	0.0051	0.0050	0.0061	...	...	0.0020	...
	3.48[a]	...	...	...	...	36.8	30.8	25.6	(2.56)	0.0050	0.0045	0.0053	...	...	...	...
	3.90	15.6	...	28.0	27.7	...	...	...	...	...	...	...	0.0038	0.0038	...	0.0021
No. 11	1.48[a]	14.1	8.0	16.5	13.2	22.8	18.5	7.4	(0.65)	0.0067	0.0078	0.0095	0.0055	0.0071	0.0033	0.0059
	1.94[a]	...	...	14.1	13.4	21.8	15.0	9.5	(1.21)	0.0051	0.0048	0.0068	0.0043	0.0046	...	...
	2.45[a]	13.5	8.9	...	...	22.5	17.2	...	...	...	0.0043	0.0056	...	...	0.0023	0.0034
	2.93[a]	...	...	...	14.2	28.3	...	...	...	...	...	0.0061	0.0030	...	...	...
	3.48[a]	...	...	19.4	...	27.7	22.0	14.5	(2.45)	0.0037	0.0038	0.0049	...	0.0042	...	...
	3.90	15.6	11.5	22.4	17.9	...	...	...	...	...	...	...	0.0029	0.0037	0.0018	0.0025
No. 9	1.48					19.6	15.8	4.5	(0.50)	0.0061	0.0082	0.0104				
	1.94					18.6	14.0	5.3	(0.96)	0.0041	0.0054	0.0072				
	2.93					21.9	18.3	...	...	...	0.0047	0.0058				
	3.48					25.0	19.1	7.9	(2.50)	0.0024	0.0041	0.0054				
No. 6	1.48					13.2	8.8	3.1	(0.78)	0.0052	0.0060	0.0090				
	1.94					13.4	11.4	5.8	(1.24)	0.0052	0.0056	0.0071				
	2.93					15.8	11.4	...	...	...	0.0038	0.0053				
	3.48					17.3	14.0	8.6	(2.78)	0.0029	0.0039	0.0049				

[a] Exposure time for Pittsburgh samples. The exposure time for Bridgeport and for Sandy Hook is from 1 per cent to 2 per cent less than the Pittsburgh time.

[b] These figures apply to the zinc-coated wires only.

[c] Diameter decrease $= \dfrac{D(1 - \sqrt{Sr})}{t}$

where: D = original wire diameter,
t = years since the beginning of rust, and
Sr = percentage of strength remaining in the specimen at the time of removal.

this relation will be remains to be determined from future data. (Compare the penetration rates in column D with those of E_1 of Table E, selecting figures to compare which relate to the same gages and which have corresponding exposure times.)

The chairman of the Wire Test Inspection Committee wishes to express his appreciation to A. P. Jahn of the Bell Telephone Laboratories for his assistance in connection with the managerial work involved in the inspection of specimens, the task of analyzing data, and the preparation of this report.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

C. D. HOCKER,
Chairman.

TABLE I.—REPORT OF INSPECTIONS OF UNFABRICATED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. (In cases of zinc coatings, the G and Y conditions are characteristic states of corrosion of zinc iron alloys in the coatings.) R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance (usually apply to wires other than zinc-coated ones). D = dark (dirt or soot) to the exclusion of a better observation. Cr = yellow from chromate treatment. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. Asterisk (*) = wires erroneously reported as having rusted at previous inspection. For explanation see text of report. Figures (as 5, 20, 50, 100) are percentages.

Coating Group	Zinc Coating, oz. per sq. ft. of surface[a]	Wire Lot	Pittsburgh (Brunot Island) Pa.				Sandy Hook, N. J.				Bridgeport, Conn.				State College, Pa.			
			2.45 yr.	2.93 yr.	3.48 yr.	3.92 yr.	2.41 yr.	2.90 yr.	3.45 yr.	3.90 yr.	2.42 yr.	2.91 yr.	3.45 yr.	3.91 yr.	2.28 yr.	2.75 yr.	3.31 yr.	3.75 yr.
0.20 to 0.30	0.27	No. 512.....	100R	100R	100R	100R	5Y 95R	100R	100R	100R	24Y 76R	100R	100R	100R	G	G	GY	GY
	0.30	No. 410.....	100R	100R	100R	100R	67Y 33R	100R	100R	100R	65Y 35R	100R	100R	100R	G	G	GY	GY
	0.28	No. 411.....	100R	100R	100R	100R	90Y 10R	100R	100R	100R	95Y 5R	100R	100R	100R	G	G	GY	GY
	0.29	No. 510.....	100R	100R	100R	100R	85Y 45R	100R	100R	100R	*95Y	100R	100R	100R	G	G	GY	GY
	0.29	No. 412.....	100R	100R	100R	100R	*GY	100R	100R	100R	*90Y	100R	100R	100R	G	G	GY	GY
Avg. 0.28	0.24	No. 511.....	100R	100R	100R	100R	39Y 61R	100R	100R	100R	79Y 21R	100R	100R	100R	G	G	GY	GY
0.25 to 0.35	0.25	No. 315.....	100R	100R	100R	100R	15Y 85R	100R	100R	100R	54Y 46R	100R	100R	100R	G	G	GY	50Y 19R
	0.28	No. 215.....	100R	100R	100R	100R	70Y 30R	1Y 99R	100R	100R	79Y 21R	2Y 98R	100R	100R	G	G	GY	20Y 13R
	0.27	No. 117.....	100R	100R	100R	100R	*GY	5Y 95R	100R	100R	100Y	43Y 57R	25Y 75R	100R	G	G	GY	GY
	0.38	No. 317.....	100R	100R	100R	100R	*GY	GY	GY	60Y 40R	100Y	90Y 10R	86Y 14R	18Y 82R	G	G	GY	GY
	0.36	No. 217.....	100R	100R	100R	100R	*GY	74Y 26R	40Y 60R	100R	100Y	48Y 52R	30Y 70R	100R	G	G	GY	GY
	0.32	No. 316.....	100R	100R	100R	100R	*GY	1Y 99R	100R	100R	100Y	16Y 84R	7Y 93R	100R	G	G	GY	GY
	0.30	No. 216.....	100R	100R	100R	100R	*GY	6Y 94R	100R	100R	100Y	67Y 33R	18Y 82R	100R	G	G	GY	GY
Avg. 0.30	0.25	No. 115.....	100R	100R	100R	100R	58Y 42R	1Y 99R	100R	100R	60Y	100R	100R	100R	G	G	GY	10Y 7R
0.35 to 0.45	0.36	No. 520.....	100R	100R	100R	100R	*GY	1Y 99R	100R	100R	GY	100R	100R	100R	G	G	GY	GY
	0.35	No. 422.....	100R	100R	100R	100R	*GY	95Y 5R	93Y 7R	100R	GY	75Y 25R	35Y 65R	100R	G	G	GY	GY
	0.31	No. 423.....	100R	100R	100R	100R	*GY	52Y 48R	28Y 72R	100R	GY	20Y 80R	17Y 83R	100R	G	G	GY	GY
	0.44	No. 522.....	100R	100R	100R	100R	*GY	98Y 2R	70Y 30R	100R	GY	99Y	97Y 3R	100R	G	G	GY	GY
	0.37	No. 282.....	100R	100R	100R	100R	GY	85Y 15R	85Y 15R	100R	GY	87Y 13R	76Y 24R	100R	G	G	GY	GY
	0.38	No. 521.....	30Y 70R	100R	100R	100R	*GY	99Y 1R	98Y 2R	100R	GY	97Y	97Y 3R	100R	G	G	GY	GY
Avg. 0.38	0.47	No. 421.....	10Y 90R	100R	100R	100R	*GY	73Y 27R	72Y 28R	100R	GY	50Y 50R	38Y 62R	100R	G	G	GY	GY
0.45 to 0.55	0.44	No. 325.....	10Y 90R	100R	100R	100R	GY	GY	Y	83Y 17R	GY	G	Y	95Y 5R	G	G	GY	GY
	0.50	No. 228.....	100R	100R	100R	100R	GY	96Y 4R	96Y 4R	39Y 61R	GY	G	Y	79Y 21R	G	G	GY	GY
	0.54	No. 329.....	85Y 15R	100R	100R	100R	GY	99Y	Y	86Y 14R	GY	G	99Y	78Y 12R	G	G	GY	GY
	0.52	No. 227.....	*50Y	100R	100R	100R	GY	GY	Y	Y	GY	G	Y	Y	G	G	GY	GY
	0.41	No. 125.....	100R	100R	100R	100R	GY	GY	99Y 1R	4Y 96R	GY	G	Y	36Y 64R	G	G	GY	GY
	0.54	No. 127.....	7Y 93R	100R	100R	100R	GY	GY	Y	99Y 1R	GY	G	Y	Y	G	G	GY	GY
	0.38	No. 326.....	100R	100R	100R	100R	GY	GY	GY	93Y 7R	GY	GY 2R	97Y 3R	76Y 24R	G	G	GY	GY
	0.45	No. 226.....	70Y 30R	100R	100R	100R	GY	GY	Y	99Y 1R	GY	G	Y	96Y 4R	G	G	GY	GY
	0.47	No. 327.....	64Y 46R	100R	100R	100R	GY	GY	Y	87Y 13R	GY	G	Y	95Y 5R	G	G	GY	GY
Avg. 0.47	0.45	No. 225.....	92Y 8R	100R	100R	100R	GY	GY	Y	99Y 1R	GY	G	Y	85Y 1R	G	G	GY	GY
0.50 to 0.60	0.61	No. 530.....	100R	100R	100R	100R	G	M 1R	MG 1R	G 1R	MG	G	G	G	MG	MG	MG	MG
	0.57	No. 432.....	100R	100R	100R	100R	MG	MG	MG	G	MG	G	G	G	MG	MG	MG	MG
	0.60	No. 531.....	100R	100R	100R	100R	G	G	GY	GY	G	G	GY	GY	MG	MG	G	G
	0.48	No. 431.....	100R	100R	100R	100R	GY	GY	Y	35Y 65R	GY	G	GY	30Y 70R	G	G	GY	GY
Avg. 0.56	0.54	No. 430.....	GY	3Y 97R	100R	100R	GY	GY	Y	Y	G	G	GY	GY	G	G	GY	GY

0.60 to 0.70	0.69	No. 338	100R	100R	100R	100R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	0.68	No. 236	100R	100R	100R	100R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	0.61	No. 335	100R	100R	100R	100R	G	G	GY	Y	G	G	GY	Y	MG	MG	G	G
	0.63	No. 237	77Y 23R	100R	100R	100R	G	G	GY	GY	G	G	Y	GY	MG	MG	G	G
	0.74	No. 135	100Y	100R	100R	100R	G	GY	GY	GY	G	G	Y	Y	MG	MG	G	G
	0.57	No. 337	*99Y	100R	100R	100R	GY	GY	Y	Y	GY	G	GY	Y	G	G	GY	GY
	0.67	No. 235	*99Y	21Y 79R	100R	100R	GY	GY	Y	Y	GY	G	Y	GY	G	G	G	G
	0.65	No. 239	100Y	100R	100R	100R	GY	GY	Y	Y	GY	G	Y	Y	G	G	GY	GY
	0.67	No. 138	98Y 2R	100R	100R	100R	GY	GY	Y	Y	GY	G	Y	Y	G	G	GY	GY
	0.64	No. 139	90Y 10R	100R	100R	100R	GY	GY	Y	Y	GY	G	Y	Y	G	G	GY	GY
	0.66	No. 336	100Y	22Y 78R	20Y 80R	100R	GY	GY	Y	Y	GY	G	GY	Y	G	G	GY	GY
Avg. 0.65	0.60	No. 238	99Y 1R	100R	100R	100R	GY	GY	Y	Y	GY	G	Y	Y	G	G	GY	GY
0.70 to 0.85	0.70	No. 540	100R	100R	100R	100R	MG	MG	MG	G	G	G	GY	G	MG	MG	MG	MG
	0.72	No. 442	100R	100R	100R	100R	MG	MG	MG	G	MG	MG		G	MG	MG	MG	MG
	0.79	No. 543	75Y 25R	100R	100R	100R	G	G	G	G	G	G	G	G	MG	MG	MG	G
	0.78	No. 441	96Y 4R	100R	100R	100R	G	MG	G	G	G	G	GY	G	MG	MG	MG	G
	0.70	No. 542	100R	100R	100R	100R	G	G	GY	GY	G	G	GY	GY	MG	MG	G	G
	0.74	No. 443	82Y 18R	100R	100R	100R	G	MG	G	G	G	G	GY	G	MG	MG	MG	G
	0.74	No. 495	80Y 20R	100R	100R	100R	G	G	G	G	Cr	G	Cr	Cr	MG	MG Cr	Cr	Cr
	0.76	No. 541	100Y	6Y 94R	1Y 99R	100R	GY	GY	GY	Y	GY	G	GY	GY	G	G	GY	G
	0.76	No. 440	100Y	100R	100R	100R	GY	GY	Y	Y	GY	G	GY	GY	G	G	GY	G
Avg. 0.75	0.87	No. 444	100Y	5Y 95R	1Y 99R	100R	GY	GY	Y	Y	GY	G	GY	GY	G	G	GY	G
0.80 to 1.00	1.05	No. 348	MG	G	40Y 60R	100R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	0.81	No. 248	100R	100R	100R	100R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	G
	0.88	No. 347	99Y	5Y 95R	100R	100R	MG	MG	G	G	G	G	G	G	MG	MG	MG	G
	0.95	No. 246	GY	GY 18R	50Y 50R	100R	MG	MG	G	G	MG	G	G	G	MG	MG	G	G
	0.88	No. 346	GY	1Y 99R	100R	100R	MG	MG	GY	GY	G	G	GY	GY	MG	MG	G	G
	0.81	No. 245	97Y 3R	100R	100R	100R	G	G	GY	Y	G	G	Y	Y	MG	MG	G	G
	0.81	No. 296	100Y	3Y 97R	100R	100R	Cr	G	G	GY	Cr	G Cr	Cr	Cr	Cr	Cr	Cr	Cr
	0.90	No. 148	GY	25Y 75R	8Y 92R	100R	G	G	GY	GY	G	G	GY	GY	MG	MG	G	G
	0.85	No. 149	GY	25Y 75R	4Y 96R	100R	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.84	No. 345	GY	11Y 89R	100R	100R	GY	GY	Y	Y	GY	G	Y	GY	G	G	GY	G
Avg. 0.88	0.96	No. 247	GY	GY 6R	94Y 6R	100R	GY	GY	Y	Y	GY	G	Y	G	G	G	GY	G
Avg. 1.28	1.35	No. 800	GY	GY	GY	98Y 2R	MG	MG	G	G	MG	G	G	G	MG	MG	MG	MG
	1.20	No. 801	GY	GY	99Y 1R	5Y 95R	MG	MG	G	G	MG	G	G	C	MG	MG	MG	MG
1.60 to 1.80	2.08	No. 552	MG	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.83	No. 451	MG	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.75	No. 350	MG	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.69	No. 252	MG	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.65	No. 351	MG	G 1R	G 2R	G 3R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.76	No. 250	MG	G	G	5Y 2R	MG	MG	MG	G	MG	G	G	G	MG	MG	MG	MG
	1.69	No. 551	MG	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.50	No. 450	MG	G 1R	25Y 2R	10Y 28R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.75	No. 352	MG	G	G	5Y 1R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	1.86	No. 251	MG	G	G	40Y 3R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
Avg. 1.76	1.86	No. 151	MG	G	G	10Y 3R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
Heavy Avg. 2.89	2.80	No. 397	MG	G	G	G	M	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	2.72	No. 298	MG	G	G	G	M	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG
	3.00	No. 299	MG	G	G	G	M	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG

[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

TABLE I.—*Continued.*

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. Cr = yellow from chromate treatment. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. Figures (as 5, 20, 50, 100) are percentages.

Coating Group	Zinc Coating, oz. per sq. ft. of surface[a]	Wire Lot	Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			3.08 yr.	4.05 yr.	2.98 yr.	4.08 yr.	2.94 yr.	3.88 yr.	3.00 yr.	3.95 yr.	2.94 yr.	3.83 yr.	2.83 yr.	3.80 yr.	2.94 yr.	3.83 yr.
0.20	0.27	No. 512	G	10Y 9R	G	G	G	G	G	G	G	G	MG	G	G	G
to	0.30	No. 410	G	15Y 1R	G	G	G	G	G	G	G	G	MG	MG	G	G
0.30	0.28	No. 411	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
Avg.	0.29	No. 510	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
0.28	0.29	No. 412	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
	0.24	No. 511	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.25	0.25	No. 315	G	20Y 15R	G	G	G	G	G	G	G	G	G	G	G	G
to	0.28	No. 215	G	20Y 1R	G	G	G	G	G	G	G	MG	G	G	G	G
0.35	0.27	No. 117	G	G	G	G	G	G	G	G	G	G	G	G	G	G
Avg.	0.38	No. 317	G	G	G	G	G	G	G	G	G	MG	MG	MG	G	G
0.30	0.36	No. 217	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.32	No. 316	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.30	No. 216	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.25	No. 115	G	10Y	G	G	G	G	G	G	G	G	MG	MG	G	G
0.35	0.36	No. 520	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
to	0.35	No. 422	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.45	0.31	No. 423	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
Avg.	0.44	No. 522	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.38	0.37	No. 282	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.38	No. 521	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.47	No. 421	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.45	0.44	No. 325	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
to	0.50	No. 228	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.55	0.54	No. 329	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
Avg.	0.52	No. 227	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.47	0.41	No. 125	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
	0.54	No. 127	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.38	No. 326	G	G	G	G	G	G	G	G	G	MG	MG	MG	G	G
	0.45	No. 226	G	G	G	G	G	G	G	G	G	G	G	G	G	G
	0.47	No. 327	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
	0.45	No. 225	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.50	0.61	No. 530	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	MG	MG	M
to	0.57	No. 432	MG	MG	MG	MG	MG	MG	M	M	MG	G	MG	MG	MG	M
0.60	0.60	No. 531	MG	G	MG	G	MG	MG	MG	MG	MG	M	M	MG	MG	MG
Avg.	0.48	No. 431	G	G	G	G	G	G	G	G	G	M	MG	MG	G	G
0.56	0.54	No. 430	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G

0.60 to 0.70 Avg. 0.65	0.69	No. 338	MG	MG	MG	MG	MG	MG	M	M	MG	M	M	M	MG	M
	0.68	No. 236	MG	MG	MG	MG	MG	MG	M	M	MG	M	M	M	MG	M
	0.61	No. 335	MG	G	MG	MG	MG	MG	MG	MG	MG	M	G	G	MG	MG
	0.63	No. 237	MG	MG	MG	MG	MG	MG	M	MG	MG	M	MG	MG	MG	M
	0.74	No. 135	MG	G	MG	G	MG	MG	MG	G	MG	M	G	G	MG	MG
	0.57	No. 337	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.67	No. 235	G	G	G	G	G	G	G	G	G	G	MG	GY	G	G
	0.65	No. 239	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.67	No. 138	G	G	G	G	G	G	G	G	G	G	MG	GY	G	G
	0.64	No. 139	G	G	G	G	G	G	G	G	G	G	MG	GY	G	G
	0.66	No. 336	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.60	No. 238	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.70 to 0.85 Avg. 0.75	0.70	No. 540	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	MG	MG	MG
	0.72	No. 442	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	MG
	0.79	No. 543	MG	MG	MG	MG	MG	MG	M	MG	MG	M	G	G	MG	MG
	0.78	No. 441	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	G	MG	MG	MG
	0.70	No. 542	MG	G	MG	G	MG	MG	M	MG	MG	M	MG	MG	MG	M
	0.74	No. 443	MG	MG	MG	MG	MG	MG	M	MG	MG	M	G	G	MG	MG
	0.74	No. 495	Cr	Cr	Cr	Cr	Cr	Cr	Cr	M	....	....	M	Cr	....	....
	0.76	No. 541	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.76	No. 440	G	G	G	G	G	G	G	G	G	G	MG	GY	G	G
	0.87	No. 444	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
0.80 to 1.00 Avg. 0.88	1.05	No. 348	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	M	M
	0.81	No. 248	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	M	M	M
	0.88	No. 347	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	G	G	MG	M
	0.95	No. 246	MG	MG	MG	MG	MG	MG	M	MG	MG	M	G	G	MG	M
	0.88	No. 346	MG	G	MG	MG	MG	MG	MG	MG	MG	M	MG	MG	MG	MG
	0.81	No. 245	MG	G	MG	MG	MG	MG	MG	MG	MG	M	MG	MG	MG	MG
	0.81	No. 296	Cr	Cr	Cr	Cr	Cr	Cr	Cr	M	....	....	....	Cr	....	....
	0.90	No. 148	MG	G	MG	G	MG	MG	MG	MG	MG	M	G	G	MG	MG
	0.85	No. 149	MG	MG	MG	G	MG	MG	M	M	MG	M	G	G	MG	M
	0.84	No. 345	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
	0.96	No. 247	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
Avg. 1.28	1.35	No. 800	MG	MG	MG	G	MG	MG	M	M	....	....	G	G	....	....
	1.20	No. 801	MG	MG	MG	G	MG	MG	M	M	....	....	G	G	....	....
1.60 to 1.80 Avg. 1.76	2.08	No. 552	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	MG
	1.83	No. 451	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	MG
	1.75	No. 350	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
	1.69	No. 252	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
	1.65	No. 351	MG	MG	MG	G	MG	MG	M	M	MG	M	G	MG	M	M
	1.76	No. 250	MG	MG	MG	G	MG	MG	MG	M	MG	M	MG	MG	MG	MG
	1.69	No. 551	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	M
	1.50	No. 450	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	MG
	1.75	No. 352	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	M
	1.86	No. 251	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	M
	1.86	No. 151	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	M
Heavy Avg. 2.89	2.80	No. 397	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	M
	2.72	No. 298	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	M
	3.00	No. 299	MG	MG	MG	MG	MG	MG	M	M	....	....	G	G	....	....

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

TABLE I.—*Continued*.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, R = rust of base metal, PHR, PHY = rust or yellowed in pinholes, B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance. D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. SY or SR = superficial Y and superficial R, respectively; asterisk (*) speckled appearance. Figures (as 5, 20, 50, 100) are percentages.

	Gage	Wire Lot	Pittsburgh (Brunot Island), Pa.				Sandy Hook, N. J.				Bridgeport, Conn.				State College, Pa.			
			2.45 yr.	2.93 yr.	3.48 yr.	3.92 yr.	2.41 yr.	2.90 yr.	3.45 yr.	3.90 yr.	2.42 yr.	2.91 yr.	3.45 yr.	3.91 yr.	2.28 yr.	2.75 yr.	3.31 yr.	3.75 yr.
Copper-covered wire	11	No. 021, 7.42-oz. coating	G	G	G	Gn	Gn	Gn	Gn	Gn	Gn	G	G	G	Gn	Gn	G	Gn
	9	No. 022, 9.24-oz. coating	G	G	G	Gn	Gn	Gn	Gn	Gn	Gn	G	G	G	Gn	Gn	G	Gn
Lead-coated wires	6	No. 040, 1.40-oz. coating	17PHR	32PHR	15PHR	25PHR	43PHR	41PHR	40PHR	27PHR	6PHR	2PHR	2PHR	3PHR	26PHR	32PHR	30PHR	40PHR
	9	No. 041, 1.48-oz. coating	18PHR	33PHR	15PHR	25PHR	47PHR	51PHR	55PHR	52PHR	8PHR	3PHR	3PHR	3PHR	31PHR	40PHR	40PHR	53PHR
	11	No. 042, 1.68-oz. coating	18PHR	35PHR	15PHR	25PHR	29PHR	34PHR	35PHR	28PHR	12PHR	5PHR	3PHR	4PHR	18PHR	17PHR	20PHR	17PHR
	14½	No. 043, 1.90-oz. coating	20PHR	37PHR	15PHR	25PHR	69PHR	77PHR	80PHR	63PHR	8PHR	2PHR	2PHR	5PHR	29PHR	26PHR	30PHR	32PHR
Corrosion-Resistant Uncoated Wires:																		
12-14% Cr, cold drawn	12	No. 001	100Y	GY	GY	GY	100Y	GY	GY	Y	G	G	G	G	MG	M	M	M
	9	No. 004	100Y	GY	GY	GY	100Y	GY	GY	Y	G	G	G	G	MG	M	M	M
	17	No. 005	100Y	GY	GY	GY	100Y	GY	GY	Y	G	G	G	G	MG	M	M	M
12-14% Cr, air quenched	13	No. 002	GY	G	GY	GY	SY	GY	GY	Y	MG	MG	MG	G	M	M	M	M
	10	No. 006	GY	G	GY	GY	SY	GY	GY	Y	MG	MG	MG	G	M	M	M	M
	18	No. 007	GY	G	GY	GY	SY	GY	GY	Y	MG	MG	MG	G	M	M	M	M
18% Cr, 8% Ni	15	No. 003	M	M	M	MG	M	M	M	M	M	M	M	MG	M	M	M	M
	12	No. 008	M	M	M	MG	M	M	M	M	M	M	M	M	M	M	M	M
	20	No. 009	M	M	M	MG	M	M	M	M	M	M	M	M	M	M	M	M
	14	No. 015	M	M	M	MG	M	M	M	M	M	M	M	M	M	M	M	M

	Gage	Wire	Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			3.08 yr.	4.05 yr.	2.98 yr.	4.08 yr.	2.94 yr.	3.88 yr.	3.00 yr.	3.95 yr.	2.94 yr.	3.83 yr.	2.83 yr.	3.80 yr.	2.94 yr.	3.83 yr.
Copper-covered wire	11	No. 021, 7.42-oz. coating	Gn	Gn	Br	Br	Br	Br	Br	B	Br	Br	Br	Br	Br	Br
	9	No. 022, 9.24-oz. coating	Gn	Gn	Br	Br	Br	Br	Br	B	Br	Br	Br	Br	Br	Br
Lead-coated wires	6	No. 040, 1.40-oz. coating	25PHR	20PHR	PHY	25PHY	20PHY	25PHY	8PHR	9PHR	10PHR	12R+Y	4PHR	6PHR	5PHR	15R+Y
	9	No. 041, 1.48-oz. coating	20PHR	15PHR	PHY	22PHY	20PHY	20PHY	13PHR	26PHR	13PHR	18R+Y	4PHR	9PHR	7PHR	7R
	11	No. 042, 1.68-oz. coating	25PHR	20PHR	PHY	24PHY	5PHY	10PHY	6PHR	8PHR	4PHR	9R+Y	1PHR	5PHR	6PHR	7R+Y
	14½	No. 043, 1.90-oz. coating	10PHR	1PHR	PHY	20PHY	G	G	4PHR	3PHR	4PHR	3R+Y	G	1PHY	4PHR	2R+Y
Corrosion-Resistant Uncoated Wires:																
12-14% Cr, cold drawn	12	No. 001	MG	M	SY*	SY*	G	G	MG	MG	SR*	29SR	SR*	SR*	SR	13SR
	9	No. 004	MG	M	SY*	SY*	SY	SY	MG	MG	SR*	28SR	SR*	SR*	SR	13SR
	17	No. 005	MG	M	SY*	SY*	SY	SY	MG	MG	SR*	29SR	SR*	SR*	SR	18SR
12-14% Cr, air quenched	13	No. 002	MG	M	SY*	SY*	MG	MG	M	M	SY*	10SR	SR*	SR*	SR*	3SR
	10	No. 006	MG	M	M	M	MG	MG	M	M	SY*	10SR	SR*	SR*	SY*	1SR
	18	No. 007	MG	M	M	M	MG	MG	M	M	SY*	17SR	SR*	SR*	10SR	5SR
18% Cr, 8% Ni	15	No. 003	M	M	M	M	M	M	M	M	M	M	M	M	M	M
	12	No. 008	M	M	M	M	M	M	M	M	M	M	M	M	M	M
	20	No. 009	M	M	M	M	M	M	M	M	M	M	M	M	M	M
	[illegible]	[illegible]	M	M	M	M	MG	M	M	M	M		M	M	M	...

[illegible] appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. RB = rust on barbs. SR = superficial rust, asterisk (*) = speckled.

Coating Group	Average Coating, oz. per sq. ft. of surface[a]	Wire[b]	State College, Pa.				Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			1.38 yr.	1.85 yr.	2.41 yr.	2.85 yr.	3.08 yr.	4.05 yr.	2.98 yr.	4.08 yr.	2.94 yr.	3.88 yr.	3.00 yr.	3.95 yr.	2.94 yr.	3.83 yr.	2.83 yr.	3.80 yr.	2.94 yr.	3.83 yr.
0.20 to 0.30	0.30	No. C410	G	G	G	GRB	GRB	30R	G	G	G	G	G	GRB	G	MG	MG	G	G	G
	0.24	No. C411	G	G	GRB	GRB	GRB	10R	G	....	G	G	G	GRB	G	GRB	MG	MG	G	G
	0.29	No. C412	G	G	G	GY	G	GRB	G	G	G	G	G	GRB	G	G	M	G	G	G
0.35 to 0.45	0.37	No. C421	G	G	G	GY	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
	0.47	No. C422	G	G	G	GY	G	GRB	G	G	G	G	G	G	G	G	MG	G	G	G
	0.31	No. C420	G	G	G	GY	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.50 to 0.60	0.48	No. C430	G	G	G	GY	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.54	No. C431	G	G	G	GY	G	GRB	G	G	G	G	G	GRB	G	G	MG	MG	MG	G
	0.57	No. C432	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
0.70 to 0.85	0.72	No. C441	MG	MG	MG	G	MG	MG	MG	MG	MG	MG	M	M	G	M	MG	G	MG	MG
	0.78	No. C442	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	G	MG	MG
	0.74	No. C443	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	MG	MG	MG	M	MG	G	MG	MG
0.80 to 1.00	0.97	No. C445	MG	MG	MG	MG	MG	G	MG	G	MG	MG	MG	MG	MG	M	MG	G	MG	MG
	0.92	No. C446	MG	MG	MG	MG	MG	G	MG	G	MG	MG	MG	MG	MG	M	M	MG	MG	MG
1.60 to 1.80	1.64	No. C450	MG	MG	MG	G	MG	G	MG	G	MG	MG	MG	MG	G	M	MG	G	MG	M
	1.50	No. C452	MG	MG	MG	G	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	MG
	1.83	No. C451	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
Heavy Coating	1.99	No. C494	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	G	MG	M
	1.99	No. C494	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	G	MG	M
	2.00	No. C447	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	MG
Copper covered		No. C025	Br	Gn	G	Gn	Br	Gn	....	....	Br	Br	M	B	Br	Br	Br	Br	Br	BrRB
(3.97 oz. per sq. ft.)		No. C025	Br	Gn	G	Gn	....	....	Br	Br	....	....	....	....	....	....	....	....	....	....
Lead coated		No. C045	....	....	....	....	30PHR	10PHR	15PHY	15PHY	15PHY	15PHY	....	2R	15PHR	17R+Y	5PHY	5PHR	10SR*	15SR*
(1.83 oz. per sq. ft.)		No. C045	Gn	Gn	G	GRB	....	....	10PHY	10PHY	....	....	10R	5R	10PHR	....	5PHY	5PHR	....	....

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

[b] These barbed wire numbers do not always correspond to the unfabricated wire numbers as seen below:

Barbed Wire	Unfabricated Wire Lot	Barbed Wire	Unfabricated Wire Lot
No. C410	No. 410	No. C441	No. 442
No. C411	....	No. C442	No. 441
No. C412	No. 412	No. C443	No. 443
No. C421	....	No. C445	....
No. C422	No. 421	No. C446	....
No. C420	No. 423	No. C447	....
No. C430	No. 431	No. C450	....
No. C431	No. 430	No. C452	No. 450
No. C432	No. 432	No. C451	No. 451
		No. C494	....

TABLE III.—REPORT OF INSPECTIONS OF FARM FIELD FENCE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. SY or SR = superficial Y and superficial R, respectively; asterisk (*) = speckled appearance.

Coating Group	Zinc Coating, oz. per sq. ft. of surface^a	Fence	State College, Pa.				Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
			1.38 yr.	1.85 yr.	2.41 yr.	2.85 yr.	3.08 yr.	4.05 yr.	2.98 yr.	4.08 yr.	2.94 yr.	3.88 yr.	3.00 yr.	3.95 yr.	2.94 yr.	3.83 yr.	2.83 yr.	3.80 yr.	2.94 yr.	3.83 yr.
0.20	0.27	No. 512	G	G	G	G	GY	25R	G	G	G	G	G	G	G	G	MG	G	G	G
to	0.30	No. 410	G	G	G	G	10R	15R	G	G	G	G	MG	G	G	MG	MG	G	MG	G
0.30	0.28	No. 411	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
Avg.	0.29	No. 510	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
0.28	0.29	No. 412	G	G	G	G	G	G	G	G	G	G	G	G	G	G	M	G	G	G
	0.24	No. 511	G	G	G	G	G	25R	G	G	G	G	G	G	G	G	MG	MG	G	G
0.25	0.25	No. 315	G	G	G	G	5R	25R	G	G	G	G	MG	G	G	MG	MG	G	G	G
to	0.28	No. 215	G	1R	1R	1R	10R	40R	G	G	G	G	1R	1R	G	MG	MG	G	G	G1R
0.35	0.38	No. 317	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
Avg.	0.32	No. 316	G	G	G	G	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
0.30	0.30	No. 216	G	G	G	G	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
0.35	0.36	No. 520	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
to	0.35	No. 422	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
0.45	0.31	No. 423	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
Avg.	0.44	No. 522	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.38	0.38	No. 521	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
	0.47	No. 421	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
0.45	0.44	No. 325	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
to	0.50	No. 228	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	GY	G	G
0.55	0.51	No. 227	G	G	G	G	G	G	G	G	G	G	G	G	G	GY	M	MG	G	G
Avg.	0.38	No. 326	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
0.46	0.47	No. 327	G	G	G	G	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
	0.45	No. 225	G	G	G	G	G	G	G	G	G	G	G	G	G	G	M	MG	G	G
0.50	0.61	No. 530	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	MG	MG	M	MG	G	MG	MG
to	0.57	No. 432	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
0.60	0.60	No. 531	MG	MG	G	G	MG	G	MG	G	MG	MG	MG	MG	MG	M	G	G	MG	MG
Avg.	0.48	No. 431	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
0.54	0.54	No. 430	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.49	No. 532	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G
	0.49	No. 433	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	G	G	G

0.60 to 0.70 Avg. 0.64	0.69	No. 338	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	MG
	0.68	No. 236	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	G	MG	MG
	0.61	No. 335	MG	MG	G	G	MG	G	MG	G	MG	MG	MG	MG	MG	M	G	G	MG	MG
	0.63	No. 237	MG	MG	G	MG	MG	G	MG	G	MG	MG	MG	MG	MG	M	G	G	MG	MG
	0.57	No. 337	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.67	No. 235	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	M	MG	G	G
	0.66	No. 336	G	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G
	0.60	No. 238	G	G	G	G	G	G	G	G	G	G	G	G	G	GY	MG	G	G	G
0.70 to 0.85 Avg. 0.74	0.70	No. 540	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	M	MG	MG	M	MG	G	MG	MG
	0.72	No. 442	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	MG
	0.79	No. 543	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	M	MG	MG	M	G	G	MG	MG
	0.78	No. 441	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	MG	G	M	G	G	MG	MG
	0.70	No. 542	MG	MG	MG	MG	MG	G	MG	G	MG	MG	M	MG	MG	M	MG	G	MG	MG
	0.74	No. 443	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	MG	MG	MG	M	MG	G	MG	MG
	0.76	No. 541	MG	MG	G	MG	MG	MG	MG	G	MG	MG	M	MG	MG	M	G	G	MG	MG
	0.76	No. 440	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	G	G	MG	MG
0.80 to 1.00 Avg. 0.90	1.05	No. 348	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	M	G	MG	MG
	0.81	No. 248	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	G	MG	MG
	0.88	No. 347	MG	MG	MG	MG	MG	MG	MG	G	MG	MG	MG	M	MG	M	G	G	MG	MG
	0.95	No. 246	MG	MG	G	G	MG	MG	MG	G	MG	MG	MG	MG	MG	M	G	G	MG	MG
	0.88	No. 346	MG	MG	G	G	MG	G	MG	G	MG	MG	MG	MG	MG	M	MG	G	MG	MG
	0.81	No. 245	MG	MG	MG	MG	MG	G	MG	G	MG	MG	MG	MG	MG	M	G	G	MG	MG
	0.84	No. 345	G	G	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G
	0.96	No. 247	MG	MG	G	G	G	G	G	G	G	G	G	G	G	MG	MG	G	G	G
1.60 to 1.80	1.75	No. 350	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	M
	1.69	No. 252	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	M	MG	MG	MG
	1.75	No. 352	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	MG
Avg. 2.76	2.80	No. 397	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	G	G	MG	M
	2.72	No. 298	MG	MG	MG	MG	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	M
12-14% Cr, cold drawn		No. 001	M	M	M	M	MG	MG	SY*	SY*	MG*	MG*	M*	MG*	80SR	25SR	15SR*	20SR	25SR*	10SR
12-14% Cr, air quenched		No. 002	M	M	M	M	M	MG	MG	MG	MG	MG	M*	MG*	75SR	10SR	5SR*	15SR	25SY*	3SR
18% Cr, 8% Ni		No. 003	M	M	M	M	M	M	M	M	M	M	M	M	M	M	2SY*	5SY	M	M
Copper covered (7.42 oz. per sq. ft.)		No. 020	Gn	3R	1R	Gn	Gn	Gn	Br	Br	Br	Br	M	B	Br	Br	Br	Br	Br	Br
Lead coated (1.48 oz. per sq. ft.)		No. 040	10PHR	30PHR	15R+Y	30R+Y	30R	40R+Y	25PHY	35PHY	20PHY	20PHY	15R	10R	20PHR	30R+Y	5PHR	10PHR	25SR*	20SR*

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[a] The zinc coating weight reported in this column is the average of all stripping test determinations made on samples of unfabricated wire, fence or barbed wire of the specific wire lot number.

TABLE IV.—REPORT OF INSPECTIONS OF CHAIN LINK SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y. Asterisk (*) = sample disappeared—probably stolen.

All 200 series wires are 9 gage. Lead-coated wire, 9 gage. Corrosion-resistant steel, 14 gage.

All 100 series wires are 6 gage. Copper-covered wire 0.128d.

Zinc Coating, oz. per sq. ft. of surface, by stripping test	Fence	Pittsburgh, Pa.				Sandy Hook, N. J.				Bridgeport, Conn.				State College, Pa,				Lafayette, Ind.		Ithaca, N. Y.		Santa Cruz, Calif.		Davis, Calif.	
		2.45 yr.	2.93 yr.	3.48 yr.	3.92 yr.	2.41 yr.	2.90 yr.	3.45 yr.	3.90 yr.	2.42 yr.	2.91 yr.	3.45 yr.	3.91 yr.	1.38 yr.	1.85 yr.	2.41 yr.	2.85 yr.	3.08 yr.	4.05 yr.	3.00 yr.	3.95 yr.	2.94 yr.	3.83 yr.	2.94 yr.	3.83 yr.
0.29	No. E280[a]	100R	100R	100R	100R	30R	95R	95R	100R	2R	50R	92R	99R	G	G	GY	GY	G	G	G	G	G	MG	G	G
0.52	No. E281	40R	100R	100R	100R	2R	5R	7R	15R	5R	5R	5R	10R	G	G	G	GY	G	G	MG	G	G	MG	G	G
0.37	No. E282	100R	100R	100R	100R	5R	50R	70R	94R	GY	5R	25R	99R	G	G	G	GY	G	G	G	G	G	MG	G	G
1.22	No. E185	GY	GY	Y	13R	G	G	G	G	MG	G	G	GY	MG	MG	G	G	MG	MG	M	MG	MG	M	MG	M
1.13	No. E285	GY	GY	Y	80R	G	G	G	G	MG	G	G	GY	MG	MG	MG	G	MG	G	M	MG	MG	M	MG	M
2.92	No. E290	MG	G	G	G	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	M
2.09	No. E291	MG	MG	G	1R	MG	MG	MG	G	MG	MG	G	G	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG
1.60	No. H280[a]	GY	5R	10R	70R	G	G	G	G	G	G	G	GY	MG	MG	MG	G	MG	MG	MG	MG	MG	M	MG	M
1.83	No. H185	GY	GY	GY	15R	G	G	G	G	G	G	G	GY	MG	MG	G	G	MG	G	MG	MG	MG	M	MG	M
1.60	No. H186	GY	GY	GY	13R	G	G	G	G	MG	G	G	GY	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	M
1.61	No. H187	GY	GY	GY	15R	G	G	G	G	MG	G	G	GY	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	M
1.55	No. H188	GY	15R	15R	75R	G	G	G	G	MG	G	G	GY	MG	MG	MG	G	MG	MG	M	MG	MG	M	MG	M
1.36	No. H285	GY	GY	GY	65R	G	G	G	G	MG	G	G	G	MG	MG	G	G	MG	G	M	M	MG	M	MG	M
1.97	No. H286	GY	GY	GY	20R	G	G	G	G	G	G	G	G	MG	MG	G	G	MG	G	MG	MG	MG	M	MG	M
1.46	No. H287	GY	1R	2R	52R	G	G	G	G	G	G	G	G	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	M
1.74	No. H288	GY	GY	GY	6R	G	G	G	G	G	G	G	G	MG	MG	MG	G	MG	MG	M	MG	MG	M	MG	MG
1.40	No. H289[b]	G	G	G	G	MG	MG	G	G	MG	MG	G	G	MG	MG	MG	G	MG	MG	MG	M	MG	M	MG	M
4.21	No. H290	G	G	GY	GY	G	G	G	G	MG	MG	G	G	MG	MG	MG	G	MG	MG	MG	M	MG	M	MG	M
Lead-coated L050 (1.75 oz. per sq. ft.)		5PHR	10PHR	10PHR	10PHR	50R+Y	40R+Y	25R+Y	25R+Y	PHY	PHY	PHY	GY	20PHY	20R+Y	30R+Y	25PHR	30PHY	20PHY	5R	2R	15PHR	15Y	5PHR	5SR*
Copper-covered K030 (8.25 oz. per sq. ft.)		G	*	....	....	Gn	Gn	Gn	Gn	Gn	G	G	Gn	Gn	Gn	G	Gn	Gn	Gn	Br	B	Gn	Br	Gn	Br
Corrosion-resistant steel J011		M	M	MG	M	M	M	M	MG	M	M	M	MG	M	M	M	M	M	M	M	M	M	M	M	M

[a] E numbers—coating applied before weaving. H numbers—coating applied after weaving.

[b] While this sample has an H number, its appearance indicates that it was galvanized before weaving.

[a] E numbers—coating applied before weaving. H numbers … indicates that it was galvanized before weaving.
[b] While this sample has an H number, its appearance indicates that it was galvanized before weaving.

TABLE V.—REPORT OF INSPECTIONS OF WIRE STRAND SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic, G = gray, Y = yellowed, or rust stained, but not showing actual rough rust of base metal. R = rust of base metal. PHR, PHY = rust or yellowed in pinholes. B, Br, Gn, respectively, signify corroded to a black, brown, or green appearance, D = dark (dirt or soot) to the exclusion of a better observation. MG = intermediate between M and G. GY = predominately gray but showing indication of Y.

Zinc Coating, oz. per sq. ft. of surface	Strand	Pittsburgh, Pa.				Sandy Hook, N. J.				Bridgeport, Conn.				State College, Pa.			
		2.45 yr.	2.93 yr.	3.48 yr.	3.92 yr.	2.41 yr.	2.90 yr.	3.45 yr.	3.90 yr.	2.42 yr.	2.91 yr.	3.45 yr.	3.91 yr.	2.28 yr.	2.75 yr.	3.31 yr.	3.75 yr.
0.69	No. D345	MG	MG	GY	20R	MG	MG	MG	G	MG	MG	MG	G	MG	MG	MG	G
0.88	No. D346	G	G	GY	1R	MG	MG	MG	G	MG	MG	MG	G	MG	MG	MG	G
0.87	No. D347	G	G	GY	10R	G	MG	G	G	MG	G	MG	G	MG	MG	MG	G
0.93	No. D348	MG	MG	G	3R	MG	MG	MG	G	MG	G	MG	G	MG	MG	MG	G
1.10	No. D360	MG	MG	MG	G	MG	MG	MG	G	MG	G	MG	G	MG	MG	MG	G
1.17	No. D361	G	G	G	GY	G	MG	G	G	MG	G	MG	G	MG	MG	MG	G
1.03	No. D362	GY	G	G	GY	G	G	G	G	MG	G	MG	G	MG	MG	MG	G
1.51	No. D365	G	G	G	GY	G	G	G	G	MG	G	MG	G	MG	MG	MG	G
1.39	No. D366	MG	MG	MG	G	MG	MG	MG	G	MG	G	MG	G	MG	MG	MG	G
1.32	No. D367	G	G	G	GY	G	G	G	G	MG	G	G	G	MG	MG	G	G
1.71	No. D371	MG	MG	MG	G	MG	MG	MG	G	MG	G	MG	G	MG	MG	MG	G
2.47	No. D375	MG	MG	MG	G	MG	MG	MG	G	MG	G	MG	G	MG	MG	MG	G
Lead Coating (1.96 oz. per sq. ft.) D045		20PHR	10PHR	10PHR	10PHR	70R+Y	80R+Y	85R+Y	30PHR	30PHR	30PHR	35PHR	50PHR	60R+Y	80R+Y	85R+Y	20PHR

Zinc Coating, oz. per sq. ft. of surface	Strand	Lafayette, Ind.		Ames, Iowa		Manhattan, Kans.		Ithaca, N. Y.		Santa Cruz, Calif.		College Station, Tex.		Davis, Calif.	
		3.08 yr.	4.05 yr.	2.98 yr.	4.08 yr.	2.94 yr.	3.88 yr.	3.00 yr.	3.95 yr.	2.94 yr.	3.83 yr.	2.83 yr.	3.80 yr.	2.94 yr.	3.83 yr.
0.69	No. D345	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
0.88	No. D346	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	M
0.87	No. D347	MG	MG	MG	G	MG	MG	M	M	MG	MG	MG	G	MG	MG
0.93	No. D348	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
1.10	No. D360	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	M
1.17	No. D361	MG	MG	MG	G	MG	MG	M	M	MG	MG	MG	MG	MG	M
1.03	No. D362	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	MG	MG	M
1.51	No. D365	MG	MG	MG	G	MG	MG	M	M	MG	MG	MG	MG	MG	MG
1.39	No. D366	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	M
1.32	No. D367	MG	MG	MG	G	MG	MG	M	M	MG	M	MG	MG	MG	M
1.71	No. D371	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	MG	MG	M
2.47	No. D375	MG	MG	MG	MG	MG	MG	M	M	MG	M	MG	G	MG	M
Lead Coating (1.96 oz. per sq. ft.) D045		50R+Y	25R	25PHY	40PHY	15PHY	25PHY	30R	45R	17R	22R+Y	15R	25R+Y	15R	32R+Y

TABLE VI.—BREAKING LOADS AND ELONGATION OF UNFABRICATED WIRE SPECIMENS.

(Tests on Unexposed Samples and Successively Removed Companion Samples.)

Uncoated Steel Wires—Low Copper Content.

Location	Exposure, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	Breading Load, lb.	Elongation in 10 in., per cent
	Wire Lot No. 501			*Wire Lot No. 400*			*Wire Lot No. 401*			*Wire Lot No. 300*			*Wire Lot No. 302*		
Unexposed	0	500	7.3	0	480	19.2	0	450	17.2	0	730	19.5	0	1100	7.5
Pittsburgh, Pa.	2.45	270	1.2	2.45	360	11.5	2.45	310	1.9	2.45	595	16.2	2.45	820	3.4
	2.93	255	0.5	2.93	325	12.3	2.93	280	10.2	2.93	560	14.9	2.93	755	2.9
	3.48	225	0.1	3.48	325	11.0	3.48	265	8.2	3.48	565	14.6	3.48	750	2.3
Sandy Hook, N. J.	2.42	375	0.8	2.42	405	15.8	2.42	375	13.4	3.45	615	14.2	3.45	860	4.2
	2.90	340	0.2	3.90	355	11.2	3.90	315	7.6	3.90	590	13.5	3.90	830	2.6
	3.45	320	0.3	....	....	....	....	....	....	....	....	....	....	....	....
Bridgeport, Conn.	2.42	395	0.6	3.91	415	16.2	3.91	370	13.9	2.42	675	17.6	2.42	900	4.7
	2.91	380	0.5	....	....	....	....	....	....	3.91	660	16.0	3.91	875	4.0
State College, Pa.	2.75	420	0.7	2.28	460	18.3	2.28	435	14.4	2.28	705	18.7	2.28	960	5.4
Ithaca, N. Y.	3.00	460	0.8	3.95	455	17.7	3.95	425	15.6	3.95	700	16.7	3.95	965	5.3
Lafayette, Ind.	3.08	390	0.9	3.08	430	17.7	3.08	400	14.9	3.08	685	18.5	3.08	950	5.7
	4.05	375	0.5	4.05	430	16.2	4.05	395	15.4	4.05	685	17.2	4.05	920	5.0
Ames, Iowa	3.02	450	0.7	4.08	450	18.1	4.08	415	15.0	4.08	690	18.1	4.08	975	5.1
Manhattan, Kans.	3.88	475	1.2	3.88	495	18.0	3.88	470	16.6	3.88	765	17.6	3.88	1055	6.6
College Station, Tex.	3.80	470	0.7	3.80	470	16.6	3.80	435	14.6	3.80	725	16.6	3.80	1005	6.1
Davis, Calif.	3.83	495	0.7	3.83	470	17.6	3.83	460	15.1	3.83	740	18.3	3.83	1020	5.9
Santa Cruz, Calif.	3.83	465	0.5	3.83	475	16.3	3.83	435	13.2	3.83	625	16.7	3.83	1020	15.1
	Wire Lot No. 200			*Wire Lot No. 202*			*Wire Lot No. 101*			*Wire Lot No. 105*					
Unexposed	0	1045	14.8	0	1640	6.3	0	2315	6.0	0	2760	5.0			
Pittsburgh, Pa.	2.93	775	13.0	2.93	1345	2.5	2.93	1980	3.8	2.93	2290	3.0			
	3.48	765	12.3	3.48	1260	3.0	3.48	1895	3.7	3.48	2305	1.9			
Sandy Hook, N. J.	2.42	905	13.0	2.42	1460	4.6	2.42	2190	4.0	2.42	2520	3.8			
	3.45	880	13.9	3.45	1485	2.8	3.45	2045	4.3	3.45	2425	3.4			
Bridgeport, Conn.	2.42	905	13.2	2.42	1465	4.9	2.91	2130	3.4	2.91	2530	2.8			
State College, Pa.	2.75	960	12.9	2.75	1580	5.0	2.75	2240	3.9	2.75	2630	3.3			
Ithaca, N. Y.	3.95	935	12.5	3.95	1515	4.8	3.95	2235	4.8	3.95	2680	4.2			
Lafayette, Ind.	3.08	920	13.7	3.08	1555	5.7	3.08	2195	4.7	3.08	2540	3.2			
Ames, Iowa	4.08	960	12.7	4.08	1570	5.1	4.08	2225	4.1	4.08	2685	3.7			
Manhattan, Kans.	3.88	1010	12.5	3.88	1630	5.0	3.88	2305	3.2	3.88	2615	3.5			
College Station, Tex.	3.80	1025	13.7	3.80	1590	4.5	3.80	2215	4.6	3.80	2610	3.6			
Davis, Calif.	3.83	1025	13.1	3.83	1580	4.3	3.83	2340	4.5	3.83	2700	4.4			
Santa Cruz, Calif.	3.83	1010	12.2	3.83	1700	3.5	3.83	2330	3.8	3.83	2660	3.7			

Uncoated Steel Wires—Copper Bearing.

	Wire Lot No. 506			*Wire Lot No. 507*			*Wire Lot No. 405*			*Wire Lot No. 406*			*Wire Lot No. 305*		
Unexposed	0	520	7.3	0	595	1.3	0	485	20.0	0	470	17.8	0	1085	8.3
Pittsburgh, Pa.	2.45	325	0.0	2.45	370	1.1	2.45	370	12.2	2.45	365	14.2	2.45	880	14.1
	2.93	285	0.4	2.93	315	0.3	2.93	325	12.4	2.93	340	13.0	3.48	820	2.9
	3.48	270	0.1	3.48	280	0.0	3.48	325	9.9	2.48	335	10.8	....	....	....
Sandy Hook, N. J.	2.42	375	0.4	2.42	410	0.4	2.42	410	13.1	2.42	400	14.8	2.90	900	4.3
	2.90	350	0.3	2.90	380	0.2	3.90	340	10.2	3.90	350	10.8	3.90	880	3.5
	3.45	330	0.3	3.45	345	0.2	....	....	....	....	....	....	....	....	....
Bridgeport, Conn.	2.42	405	0.5	2.42	475	0.4	2.91	420	14.0	2.91	420	14.9	2.42	975	5.8
	3.45	390	0.5	3.45	435	0.4	....	....	....	....	....	....	3.91	920	5.7
State College, Pa.	2.75	420	0.4	2.75	500	0.1	2.28	450	17.7	2.28	445	18.9	2.28	1010	6.6
Ithaca, N. Y.	3.00	470	0.5	3.00	560	0.3	3.95	460	7.9	3.95	430	15.5	3.95	1010	6.1
Lafayette, Ind.	3.08	410	0.6	3.08	470	0.6	3.08	425	17.4	3.08	430	18.0	3.08	975	6.6
	4.05	400	0.4	4.05	460	0.4	4.05	410	16.2	4.05	425	17.6	4.05	970	6.0
Ames, Iowa	3.02	460	0.2	3.02	540	0.3	4.08	440	16.2	4.08	445	17.3	4.08	1020	6.8
Manhattan, Kans.	3.88	490	0.9	3.88	590	0.8	3.88	495	10.6	3.88	480	17.2	3.88	1080	6.3
College Station, Tex.	3.80	475	0.4	3.80	540	0.5	3.80	475	17.3	3.80	455	16.9	3.80	1025	7.1
Davis, Calif.	3.83	505	0.4	3.83	590	0.5	3.83	475	15.9	3.83	480	19.4	3.83	1075	7.0
Santa Cruz, Calif.	3.83	475	0.3	3.83	535	0.3	3.83	480	14.3	3.83	465	16.3	3.83	1045	16.6

	Wire Lot No. 306			*Wire Lot No. 205*			*Wire Lot No. 206*			*Wire Lot No. 102*			*Wire Lot No. 106*		
Unexposed	0	710	19.8	0	1110	16.5	0	1480	11.2	0	2095	10.8	0	2400	3.3
Pittsburgh, Pa.	2.45	600	13.6	2.93	900	12.1	2.93	1215	7.1	2.93	1805	8.4	2.93	1710	9.2
	3.48	570	15.3	3.48	885	13.4	3.48	1215	6.3	3.48	2185	2.1	3.48	2170	1.7
Sandy Hook, N. J.	2.90	630	15.1	2.42	985	13.2	2.42	1320	7.7	2.42	1840	10.2	2.42	2235	2.5
	3.90	590	14.7	3.45	950	13.5	3.45	1260	7.2	3.45	1820	9.6	3.45	2140	2.5
Bridgeport, Conn.	2.42	655	18.1	2.42	1005	13.7	2.42	1340	8.9	2.91	1910	9.7	2.91	2330	1.6
	3.91	655	17.2	....	....	....	....	....	....	....	....	....	....	....	....
State College, Pa.	2.28	690	18.1	2.75	1090	12.4	2.75	1360	8.4	2.75	1930	9.9	2.75	2340	2.0
Ithaca, N. Y.	3.95	675	16.3	3.95	1010	11.4	3.95	1340	8.1	3.95	1935	9.6	3.95	2455	1.9
Lafayette, Ind.	3.08	655	19.0	3.08	1045	13.6	3.08	1340	9.0	3.08	1915	11.0	3.08	2325	2.3
	4.05	655	17.8	....	....	....	....	....	....	....	....	....	....	....	....
Ames, Iowa	4.08	670	15.6	4.08	1100	11.4	4.08	1370	9.1	4.08	1915	10.0	4.08	2450	2.0
Manhattan, Kans.	3.88	730	17.2	3.88	1090	11.6	3.88	1435	9.1	3.88	1970	10.6	3.88	2500	2.0
College Station, Tex.	3.80	715	18.0	3.80	1080	14.0	3.80	1445	9.2	3.80	1950	10.9	3.80	2375	2.3
Davis, Calif.	3.83	720	18.1	3.83	1120	12.9	3.83	1445	9.1	3.83	2040	9.0	3.83	2450	2.1
Santa Cruz, Calif.	3.83	705	17.6	3.83	1090	13.0	3.83	1465	8.9	3.83	1990	10.4	3.83	2335	2.8

TABLE VI.—*Continued*

Zinc-Coated and Lead-Coated Steel Wires.

Location	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent
	Wire Lot No. 510				*Wire Lot No. 511*				*Wire Lot No. 512*			
Unexposed	0	....	500	1.8	0	....	285	20.5	0	....	325	19.1
Pittsburgh, Pa.	2.45	1.20	430	0.0	2.45	0.60	200	10.8	2.45	0.50	225	9.6
	2.93	1.20	375	0.5	2.93	0.60	175	9.1	2.93	0.50	190	6.5
	3.48	1.20	345	0.3	3.48	0.60	160	9.6	3.48	0.50	180	3.7
Sandy Hook, N. J.	3.45	1.75	420	0.2	3.45	1.75	265	15.5	3.45	1.55	260	11.7
Bridgeport, Conn.	3.45	2.50	475	0.3	3.45	1.80	270	18.1	3.45	1.65	280	13.9
	Wire Lot No. 317				*Wire Lot No. 282*				*Wire Lot No. 040*			
Unexposed	0	....	795	14.5	0	....	1320	13.0	0	....	1790	9.5
Pittsburgh, Pa.	2.93	1.45	685	12.6	2.93	1.45	1175	10.8	3.48	0.9	1835	7.6
	3.48	1.45	675	11.1	....	....	....	....	....	....	....	....
Sandy Hook, N. J.	2.90	3.75	810	11.8	2.90	2.80	1340	13.1	3.45	0.9	1820	8.0
Bridgeport, Conn.	2.91	2.80	820	11.9	....	....	....	....	3.45	1.40	1830	9.0

Location	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent	Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent
	Wire Lot No. 410				*Wire Lot No. 411*				*Wire Lot No. 412*			
Unexposed	0	....	570	18.0	0	....	615	6.0	0	....	830	7.2
Pittsburgh, Pa.	2.93	0.65	410	9.8	2.93	0.85	515	3.1	2.93	1.25	685	2.3
	3.48	0.65	395	9.6	3.48	0.85	450	1.7	3.48	1.25	670	1.2
	....	....	....	....	....	....	....	....	....	....	....	....
Sandy Hook, N. J.	3.45	1.80	540	14.2	3.45	1.85	585	3.5	3.45	2.50	780	2.7
Bridgeport, Conn.	3.45	1.80	550	14.3	3.45	1.90	605	6.0	3.45	2.50	770	3.5
	Wire Lot No. 041				*Wire Lot No. 042*				*Wire Lot No. 043*			
Unexposed	0	....	1070	12.8	0	....	670	13.0	0	....	300	17.1
Pittsburgh, Pa.	3.48	0.90	1085	9.1	3.48	0.9	685	7.6	2.93	0.9	290	11.3
	....	....	....	....	....	....	....	....	....	....	....	....
Sandy Hook, N. J.	3.45	0.9	1075	9.3	3.45	0.9	685	10.7	2.90	0.9	300	14.1
Bridgeport, Conn.	3.45	1.40	1075	11.9	3.45	1.40	680	12.9	3.45	1.40	305	13.7

ADDITIONAL REMOVALS—FROM PITTSBURGH ONLY.

Zinc-Coated Steel Wires.

Wire Lot	Unexposed		Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent
	Breaking Load, lb.	Elongation, per cent				
No. 315	900	13.3	3.48	0.45	735	7.1
No. 316	1035	8.2	3.48	1.20	930	3.6
No. 317	795	14.5	3.48	1.45	675	11.1
No. 215	1210	14.7	3.48	0.60	1055	11.1
No. 216	1630	8.5	3.48	1.30	1450	5.7
No. 217	1080	10.8	3.48	1.05	1085	6.1
No. 115	2715	7.8	3.48	0.65	2445	3.5
No. 117	2215	10.7	3.48	0.75	2055	9.0
No. 335	1040	7.5	2.45	1.50	990	3.1
No. 336	760	17.3	2.45	2.50	760	16.3
No. 337	860	15.8	2.45	2.50	865	15.0
No. 338	1020	2.0	2.45	2.00	1000	1.2

Wire Lot	Unexposed		Exposure, yr.	First Rust, yr.	Breaking Load, lb.	Elongation in 10 in., per cent
	Breaking Load, lb.	Elongation, per cent				
No. 235	1680	7.5	2.93	2.60	1640	5.2
No. 236	1270	6.0	2.93	2.00	1290	3.1
No. 135	2660	7.2	2.45	2.50	2577	5.0
No. 138	1960	17.7	2.45	2.00	1920	16.0
No. 139	1920	14.2	2.45	1.90	1890	10.4
No. 440	590	14.8	2.45	2.50	630	16.2
No. 441	520	17.8	2.45	1.90	550	16.0
No. 442	470	13.3	2.45	2.00	435	11.5
No. 443	750	6.2	2.45	1.90	760	....
No. 444	570	15.0	2.45	2.50	570	15.7
No. 495	750	6.2	2.45	1.90	755	5.4

PLATE I
PROCEEDINGS, AM. SOC. TESTING MATS.
VOL. 41
REPORT OF COMMITTEE A-5:
WIRE INSPECTION REPORT

REFERENCE INFORMATION

On Unfabricated Wire, Barbed Wire and Fence in the Wire Inspection Report.

The Wire Test Committee responsible for planning and arranging the exposure tests of specimens covered in this report recommends that the following statement be made in all reports relating to the samples of wire and wire products under exposure tests:

> "It is emphasized that the weight of coating ranges shown under each weight group have been arbitrarily chosen and purposely held to close limitations by the Committee and do not necessarily individually represent any manufacturers' commercial product. On the other hand, the entire range of coatings covered by all weight groups as a whole are designed to include a sufficient range of coating to furnish the useful corrosion resistance data desired."

SYSTEM OF NUMBERING SPECIMENS

Zinc coated and uncoated steel specimens (all have 3-digit numbers)..................(*c*)

When the first digit is.......	5	4	3	2	1
the respective gages are:	14.5	12.5	11	9	6

The second and third digits indicate weight of zinc coating as follows:

	oz. per sq.ft.		oz. per sq.ft.
00-04 (LC)*	uncoated	50-59 (CB)	1.6 to 1.8
05-09 (CB)*	uncoated	60-64 (CB)	1.0 to 1.2
10-14 (CB)	0.20 to 0.30	65-69 (CB)	1.3 to 1.5
15-19 (CB)	0.25 to 0.35	70-74 (CB)	1.6 to 1.8
20-24 (CB)	0.35 to 0.45	75-79 (CB)	2.4 to 2.6
25-29 (CB)	0.45 to 0.55	80-84 (CB)	less than 0.50
30-34 (CB)	0.50 to 0.60	85-89 (CB)	1.2 to 1.5
35-39 (CB)	0.60 to 0.70	90-94 (CB)	2.4 to 3.0
40-44 (CB)	0.70 to 0.85	97-99 (CB)	extra heavy
		95-96 (CB)	chromated after coating

45-49 (CB).........
- 0.80 to 1.00 on farm fence and unfabricated wire.
- 0.85 to 1.00 on barbed wire.
- 0.80 to 0.95 on wire strand.

Specimens of Special Materials:
000-015: uncoated corrosion-resistant steel.
020-030: copper-covered steel.
040-050: lead-coated steel.

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* LC = low copper content wire; CB = copper bearing wire.

Reference Characters

(*a*) .....Wire lot numbers identify wire from a coil or coils of wire supposedly alike.

Wire Lots of Zinc-Coated or Bare Mild Steel	Usually Appear in the Test as shown by ✓				
	Unfab. + Wire	Int. of + Fence	T and B + of Fence	T and B of Fence	Barbed Wire
100 series	✓ (S)	....	....	....	....
200 series	✓ (S)	✓ (S)	✓ (S)	✓ (300)	....
300 series	✓ (S)	✓ (S)	....	✓ (500)	....
400 series	✓ (S)	✓ (S)	....	....	✓ (400)
(*s*)...... 500 series	✓ (S)	✓ (S)	....	....	....

(S) The specimen numbers of the unfabricated wire or fence are the same as the wire lot numbers.

(300), (400), (500): Specimens of fence numbered in the series 300 have top and bottom wires of the 200 series wire lots; fences numbered in the 500 series have top and bottom wires of the 300 series wire lots. Specimens of barbed wire (numbered in the 400 series) have wire lots of the 400 series but not necessarily of the same identical wire lot numbers.

Par. A. The zinc-coated wire lots table below shows:

(1) All the individual numbered wire lots and the various utilizations of each wire lot in the test.

(2) The results of weight of coating tests on samples cut from each source (unfabricated wire, fence interiors, fence top and bottom wires, and barbed wires).

(*b*)...... (3) The average coating for each wire lot which is the average of all the figures in the third and following columns in each row.

These average coating figures are the ones entered in other tables of this report. For example, wherever unfabricated wire sample No. 317 or farm fence sample No. 317 are listed, their indicated weights of coating are 0.38 oz. per sq.ft., and not 0.46 and 0.34, respectively.

These wire lot and coating reference data provide an indication of the uniformity of coating on any wire lot.

Par. B. The makeup of fences table below shows:

(1) The lot number of the wire used in the top and bottom line wires of each fence. (The lot number of the wire used in the interior line and stay wires of any fence is the same as the fence number.)

(2) The type of fence, that is, the combination of gages used in its fabrication as explained below:

(*d*)......				
Type of fence..........................	4	3	2	1
Gage of interior line and stay wires....	14.5	12.5	11	9
Gage of top and bottom line wires......	11	10	9	9

+ ......Abbreviations: Unfab., unfabricated wire; Int., interior line and stay wire; T and B, top and bottom line wires.

Zinc-Coated Wire Lots: Their Coating Weights and Test Specimens in Which They Are Used

(See Par. A and reference characters above for explanations) (Coating weights, oz. per sq.ft.)

Makeup of Fences

(See Par. B and reference characters above)

ZINC-COATED WIRE LOTS: THEIR COATING WEIGHTS AND TEST SPECIMENS IN WHICH THEY ARE USED
(See Par. A and reference characters above for explanations) (Coating weights, oz. per sq.ft.)

Wire Lot (*a*) (Fence)	(*b*) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(*s*)	Coating Weight Tests on Samples Cut From Fence Int.(*s*)	Barbed Wire Coating	Barbed Wire No.
No. 510(*c*)	0.29	0.29	0.29		
No. 511	0.24	0.24	0.23		
No. 512	0.27	0.27	0.27		
No. 520	0.36	0.34	0.38		
No. 521	0.38	0.40	0.36		
No. 522	0.44	0.44	0.44		
No. 530	0.61	0.59	0.62		
No. 531	0.60	0.58	0.61		
No. 532	0.49	....	0.49		
No. 540	0.70	0.72	0.68		
No. 541	0.76	0.80	0.72		
No. 542	0.70	0.69	0.71		
No. 543	0.79	0.83	0.74		
No. 551	1.69	1.69	....		
No. 552	2.08	2.08	....		
No. 410(*c*)	0.30	0.25	0.38	0.27	(410)
No. 411	0.28	0.28	0.28	....	....
No. 412	0.29	0.30	0.30	0.27	(412)
No. 421	0.47	0.49	0.49	0.44	(422)
No. 422	0.35	0.38	0.31	....	....
No. 423	0.31	0.35	0.31	0.28	(420)
No. 430	0.54	0.56	0.52	0.53	(431)
No. 431	0.48	0.49	0.48	0.46	(430)
No. 432	0.57	0.58	0.60	0.53	(432)
No. 433	0.49	....	0.49	....	....
No. 440	0.76	0.72	0.79	....	....
No. 441	0.78	0.77	0.76	0.82	(442)
No. 442	0.72	0.65	0.86	0.64	(441)
No. 443	0.74	0.74	0.78	0.70	(443)
No. 444	0.87	0.87	....	....	....
No. 450	1.50	1.48	....	1.52	(452)
No. 451	1.83	1.71	....	1.95	(451)
No. 495 Cr	Made from unfab. wire 443.				

	Unfab. Wire		Unfab. Wire
No. 115(*c*)	0.25	No. 139	0.64
No. 117	0.27	No. 148	0.90
No. 125	0.41	No. 149	0.85
No. 127	0.54	No. 151	1.86
No. 135	0.74	No. 800(6 ga.)	1.35
No. 138	0.67	No. 801(9 ga.)	1.20

Wire Lot (*a*) (Fence)	(*b*) Average Coating	Coating Weight Tests on Samples Cut From Unfab. Wire(*s*)	Fence Int.(*s*)	T and B of Fence Top	T and B of Fence Bottom	T and B of Fence No.
No. 315(*c*)	0.25	0.25	0.25	0.25	0.26	(512)
No. 316	0.32	0.32	0.31	0.32	0.33	(510)
No. 317	0.38	0.46	0.34	0.37	0.36	(511)
No. 325	0.44	0.50	0.52	0.37	0.39	(520)
No. 326	0.38	0.40	0.35	....	....	....
No. 327	0.47	0.48	0.44	0.48	0.48	(522)
No. 329	0.54	0.56	....	0.45	0.61	(521)
No. 335	0.61	0.61	0.63	0.59	0.60	(531)
No. 336	0.66	0.73	0.60	....	....	....
No. 337	0.57	0.66	0.59	0.50	0.52	(532)
No. 338	0.69	0.67	0.76	0.67	0.65	(530)
No. 345	0.84	0.83	0.97	0.76	0.79	(541)
No. 346	0.88	0.84	0.84	0.96	0.88	(542)
No. 347	0.88	0.96	1.00	0.78	0.79	(543)
No. 348	1.05	1.04	1.07	1.05	1.06	(540)
No. 350	1.75	1.73	1.76	....	....	....
No. 351	1.65	1.65	....	....	....	....
No. 352	1.75	1.82	1.68	....	....	....
No. 397	2.80	2.80	2.79	....	....	....

Wire Lot (*a*) (Fence)	(*b*) Average Coating	Unfab. Wire(*s*)	Fence(*s*) T-B Top	Fence(*s*) T-B Bottom	T and B of Fence Top	T and B of Fence Bottom	T and B of Fence No.
No. 215(*c*)	0.28	0.28	0.25	0.25	0.33	0.29	(315)
No. 216	0.28	0.31	0.30	0.31	0.27	0.29	(316)
No. 217	0.36	0.29	....	....	0.40	0.39	(317)
No. 225	0.45	0.45	0.49	0.44	0.43	0.47	(327)
No. 226	0.45	0.46	....	....	0.45	0.45	(326)
No. 227	0.51	0.59	0.48	0.49	....	....	
No. 228	0.50	0.49	0.48	0.48	0.50	0.53	(325)
No. 235	0.67	0.82	0.60	0.62	0.68	0.64	(337)
No. 236	0.68	0.66	0.68	0.73	0.69	0.65	(338)
No. 237	0.63	0.72	0.67	0.55	0.58	0.61	(335)
No. 238	0.60	0.60	0.59	0.61	0.59	0.61	(336)
No. 239	0.65	0.65	....	....	....	....	....
No. 245	0.81	0.80	0.83	0.78	0.81	0.81	(346)
No. 246	0.95	0.97	0.94	0.98	0.89	0.95	(347)
No. 247	0.96	0.94	0.95	1.00	1.01	0.91	(345)
No. 248	0.81	0.66	0.92	0.68	0.91	0.89	(348)
No. 250	1.76	1.76	....	....	....	....	....
No. 251	1.86	1.68	....	....	1.99	1.91	(352)
No. 252	1.69	1.67	1.85	1.57	....	....	(350)
No. 282	0.37	0.37	....	....	....	....	....
No. 296 Cr	Made from unfab. wire 245.						
No. 298	2.72	2.78	2.71	2.74	2.71	2.67	(397)
No. 299	3.00	3.00	....	....	....	....	....

MAKEUP OF FENCES
(See Par. B and reference characters above)

Int. Lot (Fence)	Type (*d*)	T-B Lot	Int. Lot (Fence)	Type (*d*)	T-B Lot
ZINC-COATED FENCES					
No. 510	4	No. 316	No. 410	3	....
No. 511	4	No. 317	No. 411	3	....
No. 512	4	No. 315	No. 412	3	....
No. 520	4	No. 325	No. 421	3	....
No. 521	4	No. 329	No. 422	3	....
No. 522	4	No. 327	No. 423	3	....
No. 530	4	No. 338	No. 430	3	....
No. 531	4	No. 335	No. 431	3	....
No. 532	4	No. 337	No. 432	3	....
No 540	4	No. 348	No. 433	3	....
No. 541	4	No. 345	No. 440	3	....
No. 542	4	No. 346	No. 441	3	....
No. 543	4	No. 347	No. 442	3	....
			No. 443	3	....
No. 315	2	No. 215			
No. 316	2	No. 216	No. 215	1	No. 215
No. 317	2	No. 217	No. 216	1	No. 216
No. 325	2	No. 228	No. 225	1	No. 225
No. 326	2	No. 226	No. 227	1	No. 227
No. 327	2	No. 225	No. 228	1	No. 228
No. 335	2	No. 237	No. 235	1	No. 235
No. 336	2	No. 238	No. 236	1	No. 236
No. 337	2	No. 235	No. 237	1	No. 237
No. 338	2	No. 236	No. 238	1	No. 238
No. 345	2	No. 247	No. 245	1	No. 245
No. 346	2	No. 245	No. 246	1	No. 246
No. 347	2	No. 246	No. 247	1	No. 247
No. 348	2	No. 248	No. 248	1	No. 248
No. 350	2	No. 252	No. 252	1	No. 252
No. 352	2	No. 251	No. 298	1	No. 298
No. 397	2	No. 298			
BARE-STEEL FENCES					
No. 505	4	No. 306	No. 305	2	No. 206
No. 506	4	No. 305	No. 306	2	No. 207
No. 405	3	....	No. 205	1	No. 205
No. 406	3	....	No. 206	1	No. 206
Coppered-covered steel fence No. 020			Int. No. 021	2	No. 022
Lead-coated steel fence No. 040			Int. No. 041	1	No. 041

UNCOATED CORROSION-RESISTANT STEEL FENCES

No. 001 (T - B Lot No. 001) Gages: Int. 12 ; T - B 12
No. 002 (T - B Lot No. 002) Gages: Int. 12.5; T - B 12.5
No. 003 (T - B Lot No. —) Gages: Int. 15 ; T - B 13

TABLE VII.—LOSS OF WEIGHTS OF ZINC-COATED WIRES.

(Tests on unfrabricated wires weighed and measured before exposure.)
Losses of Weight in ounces per square foot.

Gage of Wire	Group A[a]			Group B[a]			Group C[a]			Averages of Like Gages
	Wire Lot	Rack Exposure		Wire Lot	Rack Exposure		Wire Lot	Rack Exposure		
		West Side	East Side		West Side	East Side		West Side	East Side	
Pittsburgh Exposures (2.45 yr.)										
6	No. 148	0.810 0.791	0.757 0.779							0.784
9				No. 247	0.827 0.827	0.822 [b]	No. 248	[b] [b]	[b] [b]	0.825
11	No. 346	0.917 0.885	0.876 0.882	No. 345	0.810 0.804	0.807 [b]	No. 348	0.901 0.905	0.898 0.901	0.866
14½	No. 542	[b] [b]	[b] [b]	No. 541	0.811 0.806	0.771 0.806	No. 540	[b] [b]	[b] [b]	0.798
	Avg.........	0.851	0.823	Avg.........	0.814	0.801	Avg.........	0.903	0.900	
Pittsburgh Exposures (2.93 yr.)										
All Samples except Wire Lot No. 348 partly rusted. Wire Lot No. 348 showed coating loss of 0.949 and 0.966 oz. per sq. ft. for western exposure and 0.968 and 0.966 for eastern exposure; averaging 0.962 oz. per sq. ft. for the lot.										
Sandy Hook Exposures (2.41 yr.)										
6	No. 148	0.255 0.249	0.245 0.283							0.258
9				No. 247	0.309 0.302	0.299 0.307	No. 248	0.225 0.228	0.238 0.230	0.267
11	No. 346	0.267 0.261	0.204 0.359	No. 345	0.304 0.310	0.326 0.323	No. 348	0.234 0.231	0.240 0.247	0.276
14½	No. 542	0.276 0.276	0.325 0.218	No. 541	0.326 0.331	0.331 0.346	No. 540	0.248 0.248	0.258 0.258	0.287
	Avg.........	0.264	0.272	Avg.........	0.314	0.322	Avg.........	0.236	0.245	
Sandy Hook Exposures (3.45 yr.)										
6	No. 148	0.347 0.340	0.369 0.381							0.359
9				No. 247	0.437 0.431	0.428 0.434	No. 248	0.338 0.336	0.354 0.342	0.387
11	No. 346	0.386 0.381	0.393 0.392	No. 345	0.440 0.434	0.446 0.436	No. 348	0.333 0.346	0.349 0.353	0.391
14½	No. 542	0.394 0.403	0.411 0.415	No. 541	0.461 0.444	0.466 0.473	No. 540	0.367 0.367	0.368 0.364	0.408
	Avg.........	0.375	0.393	Avg.........	0.441	0.447	Avg.........	0.348	0.355	
State College Exposures (3.31 yr.)										
6	No. 148	0.186 0.194	0.200 0.194							0.193
9				No. 247	0.207 0.204	0.203 0.221	No. 248	0.165 0.158	0.161 0.163	0.185
11	No. 346	0.195 0.195	0.185 0.205	No. 345	0.209 0.201	0.209 0.206	No. 348	0.158 0.174	0.153 0.156	0.187
14½	No. 542	0.206 0.192	0.196 0.196	No. 541	0.189 0.198	0.191 0.188	No. 540	0.171 0.132	0.160 0.154	0.181
	Avg.........	0.195	0.196	Avg.........	0.201	0.203	Avg.........	0.160	0.158	

[a] By reference to the published referee tests results, the wires listed under group A, (or B or C) will be found to be closely alike as regards base metal composition and coating characteristics.

[b] Samples partly rusted, not included in the averages.

REPORT OF COMMITTEE A-6

ON

MAGNETIC PROPERTIES

Committee A-6 on Magnetic Properties has held one meeting since its previous report: in Washington, D. C., on March 3, 1941, in conjunction with the spring group meetings.

As suggested in the 1940 report, Subcommittee V on Magnetic Analysis has been discharged due to inactivity and the apparent lack of need for work on the part of the Society in this field. If any magnetic analysis problems arise in the future, the main committee is prepared to consider them.

The memberhip of Mr. N. A. Kurman of the Kurman Electric Co. has been discontinued, due to his lack of interest as the result of inactivity in the field of magnetic analysis.

Mr. T. Zuschlag has been appointed a member of Sucommittee IV.

I. Revision of Standard

The committee recommends for immediate adoption the revision in the Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 39)[1] indicated below, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society. This revision is being recommended in view of the fact that the Modified Isthmus Method listed in the methods is now obsolete and that the High-H permeameter recently developed at the National Bureau of Standards gives satisfactory results.

Section 7 (d).—Substitute the following for the present paragraph relating to the "Modified Isthmus Method:"

> *High-H Permeameter.*—This method as described in National Bureau of Standards *Journal of Research*, Vol. 23, p. 415 (1939) is suitable for tests at magnetizing forces from 100 to 5000 oersteds.

II. Adoption of Tentative Standard as Standard

Since no adverse criticisms have been received the committee recommends that the Tentative Method of Test for Measuring Interlamination Resistance and Lamination Factor of Iron and Steel (A 34 - 40 T)[2] be approved without change for reference to letter ballot of the Society for adoption as standard. This method, when adopted, will be added to the Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 39).

III. Adoption as Standard of Tentative Revision of Standard

The committee recommends that the tentative revision[3] of the Standard Definitions of Terms, with Units and Symbols, Relating to Magnetic Testing (A 127 - 39)[4] be approved without change for reference to letter ballot of the Society for adoption as standard.

These recommendations have been submitted to letter ballot of the committee which consists of 24 members; 22 members returned their ballots, with the results shown in Table I.

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 516.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 307.

[3] 1939 Book of A.S.T.M. Standards, Part I, p. 1274 or 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 477.

[4] 1939 Book of A.S.T.M. Standards, Part I, p. 527.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF STANDARD			
Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 39), immediate adoption	22	0	0
II. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Method of Test for Measuring Interlamination Resistance and Lamination Factor of Iron and Steel (A 34 - 40 T)	21	0	1
III. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Definitions of Terms, with Units and Symbols, Relating to Magnetic Testing (A 127 - 39)	22	0	0

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Direct Current Test Methods (B. M. Smith, chairman).—As a means of obtaining an absolute check on the accuracy of permeameters for use with high coercive force materials, such as Alnico, a link sample of this material was prepared by the General Electric Co. This material had a coercivity of over 400 oersteds. The link was checked as a ring by the National Bureau of Standards and the General Electric Co. up to 1000 oersteds. The link was then cut to give two straight bars which have been tested in various permeameters by the General Electric Co., the National Bureau of Standards, the Simonds Saw and Steel Co., and Mr. F. P. Fahy up to 1000 and 2000 oersteds. The check results are remarkably close in general, indicating that tests on these materials with certain approved commercial permeameters may be made with confidence. A little later it is expected that the limits of accuracy of these permeameters may be specified as a result of these tests.

A method for testing low permeability materials was submitted by Mr. B. M. Smith of the General Electric Co. A few minor changes were suggested, and it is hoped that a detailed proposed method can soon be prepared.

Subcommittee IV on Alternating Current Test Methods (J. P. Barton, chairman).—As mentioned in the previous report, two sets of interlaboratory tests on 25 cm. Epstein test specimens are now in progress. One set, initiated by the Westinghouse Research Laboratories and tested there, has also been completed by the American Rolling Mill Co. and the Carnegie-Illinois Steel Corp. These specimens are now at the General Electric Co. Tests are being made for core loss and alternating-current permeability with double lapped joints and for core loss with butt joints. Ballistic tests are also being made with double lapped joints in the 25 cm. test frame and with the Fahy simplex permeameter. In general the test results check fairly well, but there are some discrepancies which need to be ironed out. If sufficiently close checks are obtained between the alternating-current permeability tests and the ballistic tests, it is possible that ultimately the ballistic tests may be eliminated in the commercial testing of laminated materials.

The second series of interlaboratory test specimens has to do with incremental permeability tests, which are also being made on 25 cm. Epstein specimens. Magnetic properties of laminated materials under the combined influence of alternating-current and direct-current magnetizing forces are of importance in connection with materials for use in certain radio and amplifier applications and for transformers used in certain rectifier circuits. The teeth of rotating machines are also subject to similar conditions. These test specimens were initiated by the Bell Telephone Laboratories, Inc., and have been tested by The Western Union Telegraph Co. They are now at the Westinghouse Electric and Manufacturing Co. The checks so far obtained have been very good, and the test procedure seems to be fairly well established. The chief difficulty in the procedure resulted from the change in magnetic properties with time, following the demagnetizing process when testing at low inductions.

This report has been submitted to letter ballot of the committee which consists of 24 members; 22 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

THOMAS SPOONER,
Chairman.

R. L. SANFORD,
Secretary.

REPORT OF COMMITTEE A-9
ON
FERRO-ALLOYS

Committee A-9 on Ferro-Alloys has not held a meeting since its last annual report to the Society in 1939.

Subcommittee I on Specifications (E. A. Lucas, chairman) has continued to make some progress in the preparation of specifications for silicomanganese, ferrotitanium, and ferroboron. Unfortunately, this work has not reached the point where the subcommittee is prepared to present these specifications for acceptance as tentative to the Society this year.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The Tentative Specifications for Spiegeleisen (A 98 – 39 T)[1] and the Tentative Specifications for Ferrochromium (A 101 – 39 T)[1] have been published since 1939 without revision. It accordingly seemed desirable that they be adopted as standard and a ballot was conducted in the committee on the recommendation that they be referred to letter ballot of the Society for adoption. The letter ballot of the committee which consists of 27 members resulted as follows: 27 members returned their ballots with the following results: On Tentative Specifications A 98, 25 affirmative, 0 negative, and 2 ballots marked "not voting"; on Tentative Specifications A 101, 26 affirmative, and 1 negative.

Since there has been no opportunity to consider the negative viewpoint at a meeting, only the adoption of the Tentative Specifications for Spiegeleisen (A 98 – 39 T) on which the vote was unanimous can be considered at this time, unless the negative viewpoint can be clarified at a meeting of the committee held during the annual meeting.

This report has been submitted to letter ballot of the committee which consists of 27 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

CHARLES MCKNIGHT,
Chairman.

C. M. LOEB, JR.
Secretary.

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[1] 1939 Book of A.S.T.M. Standards, Part I, pp. 988, 986.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held two meetings during the year: in Atlantic City, N. J., on June 24, 1940, during the annual meeting of the Society, and in Washington, D. C., on March 3, 1941, during the spring group meetings.

Several changes in committee personnel have occurred during the year including one resignation with several changes in committee member representation. The present membership is 80 of whom 34 are classified as producers, 34 as consumers, and 12 as general interest members.

The committee wishes to record its sorrow in the loss by death of Emery L. Lasier.

Mr. B. H. DeLong has been appointed to represent Committee A-10 on the recently organized Subcommittee on Definitions of Terms Relating to Heat Treatment of Committee E-8 on Nomenclature and Definitions. This subcommittee will cooperate with the Joint Committee on Definitions of Terms Relating to Heat Treatment consisting of representatives of the American Society for Metals, Society of Automotive Engineers, American Foundrymen's Association, and A.S.T.M., recently reorganized under the chairmanship of H. S. Rawdon, National Bureau of Standards.

The preparation of specifications for bars and wire has been postponed while a specification for seamless stainless tubes for general corrosion service is in preparation. The standard specifications for stainless steel castings are ultimately to be revised and improved when the proper information on physical properties is available.

Subsequent to the 1940 annual meeting, Committee A-10 presented to the Society through Committee E-10 on Standards new Tentative Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels. These specifications were accepted[1] by Committee E-10 on August 28, 1940, and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 280, bearing the designation A 240 - 40 T.

I. Revisions of Standards

The committee recommends for immediate adoption, revisions in the following specifications and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (*A 176 - 39*):[2]

Table I.—In order to bring these specifications into accord with present commercial practice, change the requirement for maximum manganese content for Type Nos. 430, 442, and 446 from "0.50 per cent" to "1.00 per cent."

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee A-10 reported results of the letter ballot vote as follows: Of a total membership of 81, 43 members returned their ballots, of whom 35 voted affirmatively, 1 negatively, and 7 members marked their ballots "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 374.

Also change the requirement for maximum silicon content for these types from "0.75 per cent" to "1.00 per cent."

Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet Strip, and Plate (A 167 – 39),[3] *Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (A 176 – 39),*[3] *High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 39):*[4]

In order to bring these specifications into agreement with the Tentative Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 40 T), change the term "yield point" to read "yield strength" wherever it occurs in these three specifications; also add the following as a footnote to the table of physical test requirements in each of the specifications:

> Yield strength shall be defined as the stress causing 0.5 per cent extension under load.

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard with a revision in one as indicated below:

Tentative Recommended Practice for Conducting Plant Corrosion Tests (A 224 – 39 T),[5] revised as follows:

Section 5.—Add a Note following Section 5 to read as follows:

> NOTE.—If quantitative results are desired, the number of specimens should be increased to four or five so that statistical studies can be made.

Tentative Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 40 T),[6] without revision.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 80 members; 54 members have returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISIONS OF STANDARDS			
Spec. for Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (A 176 – 39), immediate adoption	48	0	6
Spec. for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip and Plate (A 167 – 39), immediate adoption	47	1	6
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Recommended Practice for Conducting Plant Corrosion Tests (A 224 – 39 T), as revised	46	0	8
Spec. for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 40 T)	44	0	10

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (E. F. Cone, chairman; Russell Franks, secretary).—The data collected by the eight sub-groups on the various types of stainless steels are in the final stages of compilation, this work being undertaken by Russell Franks and F. L. LaQue with assistance from Society Headquarters. Publication can be expected in the near future, certainly before the end of the year.

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[3] 1939 Book of A.S.T.M. Standards, Part I, pp. 363, 374.

[4] Committee A-10 by action at its meeting on June 25, 1941, decided to rescind its recommendation as regards the proposed revision in Standard Specifications A 177 – 39, pending further investigation of the determination of yield point in cold-worked metal. This action was subsequently submitted to letter ballot of the committee which consists of 80 members; 54 members returned their ballots of whom 47 have voted affirmatively, 3 negatively, and 4 members marked their ballots "not voting."

[5] 1939 Book of A.S.T.M. Standards, Part I, p. 909.

[6] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 280.

Subcommittee IV on Methods of Corrosion Testing (E. S. Taylerson, chairman).—The Tentative Recommended Practice for Conducting Plant Corrosion Tests (A 224 – 39 T),[5] with a minor revision, has been approved by the subcommittee for adoption as standard as indicated earlier in this report. The extensive atmospheric exposure tests on wire products, under the jurisdiction of Committee A-5 on Corrosion of Iron and Steel,[7] are continuing with Committee A-10 as an interested observer. Mr. C. C. Snyder is continuing to cooperate with Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys as the representative of Committee A-10 in connection with the galvanic couple tests being made by this committee between stainless steels and other metals. Work is still proceeding in the preparation of a recommended practice for a general boiling liquid test without respect to any particular medium.

Subcommittee V on Mechanical Tests (V. N. Krivobok, chairman).—This subcommittee has been relatively inactive for some time. The subcommittee has, however, been instrumental in securing a paper by Russell Franks and W. O. Binder on "The Stress-Strain Characteristics of Cold-Rolled Austenitic Stainless Steels in Compression as Determined by the Cylinder Test Method"[8] which is to be presented at the annual meeting. This paper is a contribution from the Union Carbide and Carbon Co., Research Laboratories.

Subcommittee VI on Metallography (Russell Franks, chairman).—Work has been continued on correlating the results of metallographic examination of the 18 per cent chromium, 8 per cent nickel type alloy steel after various heat treatments using the Murakami's etch and the cyanide etch with results of boiling copper sulfate tests to determine sensitivity toward intercrystalline attack. A report on these observations will be issued shortly by the subcommittee.

Subcommittee VII on Welding (L. C. Bibber, chairman).—Contact has been maintained with the American Welding Society committees and the Engineering Research Foundation committees involved in the welding of the stainless steels in order to provide the necessary liaison between Committee A-10 and those committees.

Subcommittee VIII on Specifications for Wrought Products (F. B. Foley, chairman).—This subcommittee has been relatively inactive during the past year due to lack of demand for specifications for bars and forgings. Consideration is still being given to the formulation of specifications covering wire products.

Subcommittee IX on Specifications for Flat Products (C. C. Snyder, chairman). —As mentioned earlier in this report, the subcommittee has prepared a revision in the Standard Specifications for Corrosion-Resisting Chromium Steel Sheet, Strip, and Plate (A 176 – 39)[2] increasing the manganese and silicon contents for types 430, 442, and 446 to 1 per cent maximum so that the chemical composition requirements of these several types will be in accord with current commercial practice. The substitution of "yield strength" in place of "yield point" in the specifications for wrought stainless steel strip, sheet, and plate, as mentioned earlier in this report, is being made on the recommendation of this subcommittee.

During the year discussions were had with representatives of the Technical Committee of the Dairy Industries Supply Association and the International Association of Milk Sanitarians relative to standardizing the quality of the various surface finishes supplied on stainless steel products, particularly with respect to the development of definite standards for use in distinguishing between the various finishes. Inasmuch as no accepted method of measuring finish is available, it is not possible to include in the A.S.T.M. specifications, a description of finish that could be designated numerically and be readily interpreted by inspectors in a uniform manner. Whenever measuring means of suitable simplicity and reliability are available, Committee A-10 can again take up this question. In the meantime, the best means of assuring products meeting requirements of the dairy industry is by comparison with standard samples and the providing of these standards can best be arranged for by the Stainless Steel Technical Committee.

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[7] See p. 101.
[8] See p. 629.

The Tentative Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 – 40 T), completed during the past year, were prepared as a result of a request from the A.S.M.E. Boiler Code Committee. These specifications have been approved by the Boiler Code Committee and included in the Materials Section of the Boiler Construction Code.

Subcommittee X on Specifications for Castings (W. J. Jeffries, chairman).—Some overlapping of the work of this subcommittee with that of Subcommittee XII on Valves, Fittings, Piping and Flanges for High-Temperature Service, of Committee A-1 on Steel, has occurred as exemplified in the Standard Specifications for Chromium-Nickel Alloy-Steel Castings (A 222 – 39). It is expected that some revision of the several specifications for castings under the jurisdiction of Subcommittee X will be made following a joint committee discussion and appraisal to be made by representatives from the American Petroleum Institute, the Alloy Castings Institute, and the A.S.T.M.

Subcommittee XI on Specifications for Tubular Products (Newell Hamilton, chairman).—The subcommittee is working on specifications for stainless steel tubes for general corrosion-resisting applications. Specifications for heat-resisting alloy tubes have been already provided by Committee A-1, namely, the Tentative Specifications for Seamless Alloy-Steel Pipe for Service at Temperatures from 750 to 1100 F. (A 158 – 40 T), Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 41 T), and the Standard Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 40), in which stainless grades are included.

Committee A-10 has been instrumental in securing the paper by H. Pray, R. S. Peoples, and F. W. Fink on "Addition of Bismuth for Producing Free-Machining Stainless Steels"[9] to be presented at this annual meeting, which describes investigations conducted at Battelle Memorial Institute as part of the research sponsored by the Alloy Castings Institute.

This report has been submitted to letter ballot of the committee which consists of 80 members; 54 members returned their ballots of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

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[9] See p. 646.

REPORT OF THE RESEARCH COMMITTEE ON FATIGUE OF METALS

The Research Committee on Fatigue of Metals held a meeting in connection with the spring group meetings in Washington, D. C., on March 5, 1941, and reviewed the first draft of a report on the investigation of The Effect of Type of Testing Machine on Fatigue Test Results. At the meeting, various changes were suggested. The report of this investigation is presented as an Appendix to this report.

During the year, the cooperative editorial arrangement with *Metals and Alloys* has been continued whereby this committee furnishes editorial service for abstracts of articles on fatigue of metals.

The committee has received an assignment of funds for the purpose of carrying on a study of size effect on fatigue strength. If possible, a study of the effect of residual stresses in metals is also to be considered. Plans for carrying out this investigation were discussed at the Washington meeting, and are being more definitely formulated at the present time.

Respectfully submitted on behalf of the committee,

H. F. MOORE,
Chairman.

APPENDIX

THE EFFECT OF TYPE OF TESTING MACHINE ON FATIGUE TEST RESULTS

The object of this investigation was to study the effect of different types of fatigue testing machines on the endurance limit of metals and on the shape of the *S-N* (stress-cycle) diagram.

Types of Fatigue Testing Machines Studied

The following types of fatigue testing machines were studied:

1. Rotating-Beam Machine.—Machines of the Sondericker-Ono type with two equal loads spaced symmetrically along the length of a simple rotating beam loaded by means of dead weights were used. The machines used in this investigation were R. R. Moore machines (designated R3).

2. Rotating-Cantilever-Beam Machines.—In two of the cooperating laboratories, McAdam machines (designated R4) were used. In these machines the specimen is a rotating-cantilever beam loaded at the free end with dead weights. In another cooperating laboratory a Krouse rotating-cantilever machine (R1) was used for one series of tests, and a Boone-Wishart rotating-cantilever machine (R2) was used for a second series. In both these machines the specimen is loaded at the free end through a lever with a movable weight sliding along it.

3. Vibratory Flexural Machines.—In one of the cooperating laboratories a DeForest "Rayflex" machine (V1) was used. In this machine the specimen is a long strip or bar of metal, supported at the nodes on V-supports for free vibration. The specimen is put into vibration by a variable frequency alternating current magnet, tuned to the natural period of vibration of the specimen. Stress is controlled by varying the current in the alternating-current magnet, and stress is determined from the amplitude of vibration of the specimen, which in turn, is measured by viewing the vibrating specimen through a microscope with micrometer eyepiece. The specimen is its own dynamometer.

In another cooperating laboratory a Krouse fatigue machine for plates (V2) was used for one series of tests and a Moore-Shafer vibratory flexural machine (V3) was used for another series. In both these machines the specimen is vibrated as a cantilever beam by means of a variable-throw crank and a connecting rod. The deflection of the specimen is measured by a micrometer dial gage as the machine is turned over by hand, and each specimen before it is tested is calibrated as its own dynamometer by noting the deflection of the specimen under dead weights.

In one of the cooperating laboratories, a Howell-Howarth fixed-cantilever type of machine (FC) was used. In this machine the specimen acts as the cantilever fastened at one end. It remains stationary during the test. The other end of the specimen is deflected a measured amount by putting a pivoted bearing in a rotating head a definite amount off center. The specimen is calibrated by dead weights for deflection and is thus its own dynamometer. This type of machine differs from the vibratory type in that as the free end of the cantilever is rotated in the circle, *all* the longitudinal fibers are subjected to reversed stress.

4. Direct Axial Stress Machine (Tension-Compression).—One Haigh machine (A1) was used. This machine is fitted with a pair of opposed two-phase alternating-current magnets which pull an armature first toward one magnet and then toward the other. One end of the specimen is attached to this armature and the other to the framework of the testing machine. Stress is controlled by varying the current flowing in the magnets, and load (and consequently stress) is measured by the electromotive force induced in auxiliary coils wound on the magnet cores.

One Moore-Krouse machine (A2) was used. This machine applies pulsating (zero-maximum) tensile stress by means of a lever driven by a variable-throw cam, and steady compressive load is superimposed on the pulsating load by means of a helical spring, thus making possible any ratio from −1 to +1 of maximum to minimum load during a cycle of stress. The load (and consequently the stress) is measured by the deflection of an elastic ring to which the specimen is attached.

In one cooperating laboratory, a Templin axial-stress fatigue machine (A3) was used. In this machine the specimen is alternately stretched and compressed by means of a scotch yoke driven by a variable-throw cam. The load applied is measured by the deflection of an elastic "loop" of steel in series with the specimens. Range of stress can be adjusted by nuts acting to place a variable amount of initial load on the specimen. Each machine can apply cycles of stress to two specimens at the same time.

Of the various types of machines noted, the rotating-beam machines are "constant-stress" machines; that is, the load or moment on the specimen remains constant. The vibratory machines are "constant-strain" machines; that is, the deflection of the specimen remains constant, or very nearly so. The Haigh, the Moore-Krouse, and the Templin tension-compression machines are constant-stress machines if the load-reading devices are read at intervals during a test and the load adjusted to the desired constant value as may be found necessary.

In all tests herein reported, the cycle of stress used was a complete reversal from a maximum tensile stress to a compressive stress of equal magnitude.

METALS TESTED

Steels.—Two steels were chosen for testing, representing two important types of steel: A high-strength, heat-treated alloy steel, and a medium-strength steel. They were selected with a view of getting as nearly uniform material as possible. Each steel was furnished in the form of round bars. About 250 ft. of each steel was obtained, and the whole length rolled from one billet. Brinell hardness tests were made on a specimen from each bar of steel received.

A lot of heat-treated chromium-molybdenum steel (S.A.E. 4340) was furnished by the Timken Roller Bearing Co. This material was furnished in the form of 25 bars $\frac{3}{4}$ in. in diameter and 10 ft. long, all rolled from the same billet. These 10-ft. bars were then cut into 2-ft. lengths and heat treated, each 2-ft. length being given an identifying mark. The chemical composition, the heat treatment, and the ordinary tension test results are given in Table I. A Brinell hardness determination was made on each of the twenty-five 10-ft. bars at the University of Illinois. Brinell hardness numbers ranged from 298 to 335, averaging 322.

TABLE I.—CHEMICAL COMPOSITION, HEAT TREATMENT, AND MECHANICAL TEST PROPERTIES OF METALS TESTED.

(Data furnished by the companies supplying the metals.)

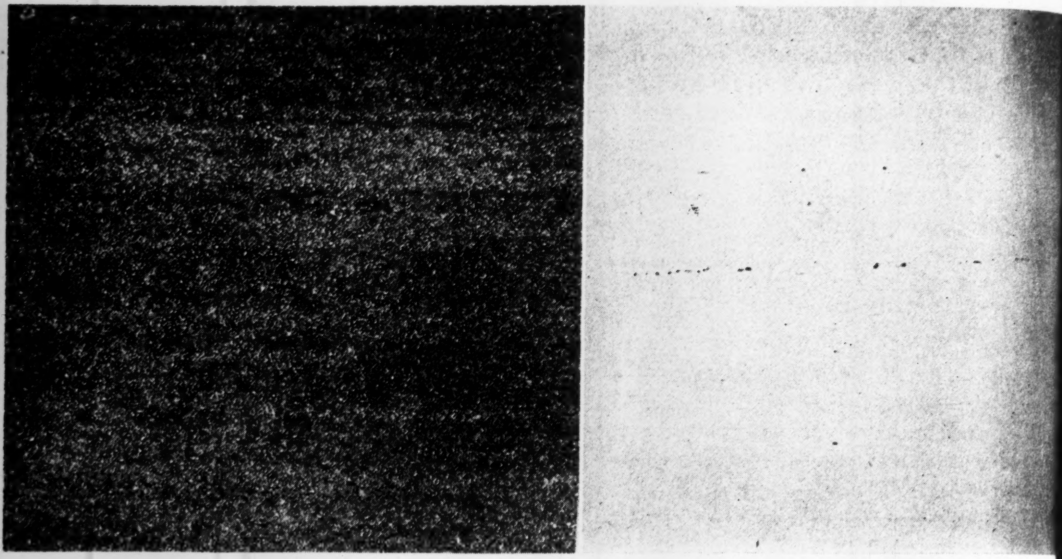
Type Number	SAE 4340	HT-50	17S-T
	Heat-treated chrome-molybdenum steel, furnished by Timken Roller Bearing Co., Canton, Ohio.	Low-alloy steel, furnished by American Rolling Mill Co., Middletown, Ohio.	Duralumin,[a] furnished by Aluminum Company of America, New Kensington, Pa.
CHEMICAL COMPOSITION, PER CENT			
Carbon	0.39	0.08	....
Manganese	0.66	0.37	0.4 to 1.0
Magnesium	....	....	0.2 to 0.75
Phosphorus	0.012	0.07	....
Sulfur	0.018	0.015	....
Silicon, max.	....	0.066	0.75
Nickel	1.72	0.69	....
Chromium, max.	0.72	....	0.25
Molybdenum	0.35	0.071	....
Iron, max.	....	....	1.0
Aluminum	....	....	remainder
Copper	....	....	3.5 to 4.5
Other elements, max.:			
Each	....	....	0.03
Total	....	....	0.10
HEAT TREATMENT			
	Normalized at 1600 F.; oil quenched from 1500 to 1525 F. Drawn at 1150 F. Cooled in air.	Tested as rolled. Finished hot.	Roll straightened after heat treatment.
MECHANICAL TEST PROPERTIES[b]			
Yield point (drop of beam), psi	142 200	49 600	....
Yield strength (0.2 per cent offset), psi	133 100	48 600	37 500
Tensile strength, psi	150 400	66 500	59 900
Elongation in 2 in., per cent	18.2	32.0	26.0
Reduction of area, per cent	61	67	38
Brinell hardness number	322	135	95
Rate of strain:			
Up to yield point, per cent per min.	0.15	0.13	....
Beyond yield point, per cent per min.	43	54	....

[a] This metal conforms to the Tentative Specifications for Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (A.S.T.M. Designation: B 89), see 1939 Book of A.S.T.M. Standards, Part I, p. 1018.

[b] Tests on SAE 4340 and HT-50 made in Talbot Laboratory, University of Illinois, Urbana, Ill. Tests on 17S-T made in Aluminum Research Laboratories, New Kensington, Pa.

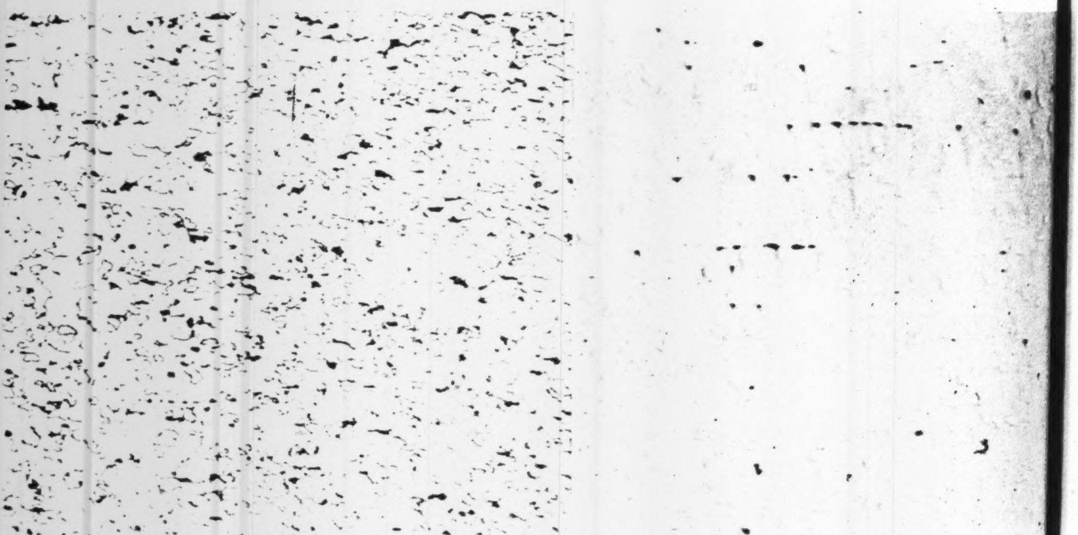
The second lot of steel was a low-alloy steel furnished by the American Rolling Mill Co. and designated by them as HT-50. This material was furnished in the form of 13 bars, each $\frac{7}{8}$ in. in diameter and approximately 19 ft. in length. Each bar was given an identifying mark. The chemical composition and the ordinary tension test results are given in Table I. This steel was tested in the "as-rolled" condition. A Brinell hardness determination was made on each of the 19-ft. bars at the University of Illinois. The Brinell hardness numbers ranged from 132 to 145, averaging 135.

Figure 1(*a*) shows the crystalline structure of the steels as shown by specimens cut transversely to the axis of the bar of steel. Figure 1(*b*) shows micrographs of unetched specimens. It



(*a*) (*b*)

S.A.E. 4340



(*a*) (*b*)

HT-50

FIG. 1.—Micrographs of Heat-Treated Alloy Steel (S.A.E. 4340) (Upper) and of Low-Alloy Steel (HT- (Lower) (× 100).

(*a*) Etched specimens showing crystalline structure.
(*b*) Unetched specimens showing inclusions.

is noted that the specimen of the low-alloy steel HT-50 shows more inclusions than does the specimen of the alloy steel S.A.E. 4340.

Duralumin.—For studying the effect of types of testing machines in fatigue tests of non-ferrous metals, duralumin was selected as the metal for test and the data obtained some time ago in the laboratories of the Aluminum Company of America by G. W. Stickley and F. M. Howell were made available to the investigation. The metal used was a lot of $\frac{3}{4}$-in. diameter rod which met the requirements of the A.S.T.M. Tentative Specifications for Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum - Copper - Magnesium - Manganese) (B 89 - 39 T).[1] Tensile properties of this metal are given in Table I.

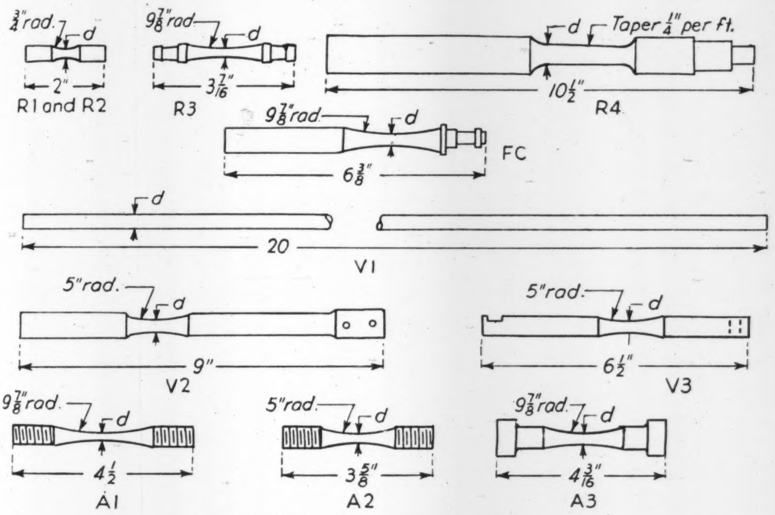


FIG. 2.—Test Specimens.
Letters and numbers (R2, V3, A1 etc.) designate the particular testing machine used. See Table II.

Specimens:

Figure 2 shows the specimens for the different testing machines studied. The drawings of the specimens are approximately to scale. Some of the specimens were machined from the bars by machine shops in Urbana, Ill.; some were machined by the cooperating laboratory (including all the duralumin specimens); and some were machined in the shop of the Talbot Materials Testing Laboratory at the University of Illinois. With all the specimens, care was taken to see that the specimens were not overheated during testing. The specimens machined in Urbana were either machined under a stream of noncorrosive coolant, or on each specimen one light cut was removed and then the specimen was set aside and a second specimen put in the lathe and a light cut taken from it, and so on. In this way no specimen became so hot that it could not be handled with the bare hand. Specimens for the vibratory tests in the Krouse and the Moore-Shafer machines were either milled flat under a stream of coolant or machined on a shaper so that they did not heat sufficiently to make handling with the bare hand difficult.

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 1018.

All the specimens were given the final polish in the Talbot Laboratory shops. All specimens were polished by one man. Each round specimen was rotated slowly

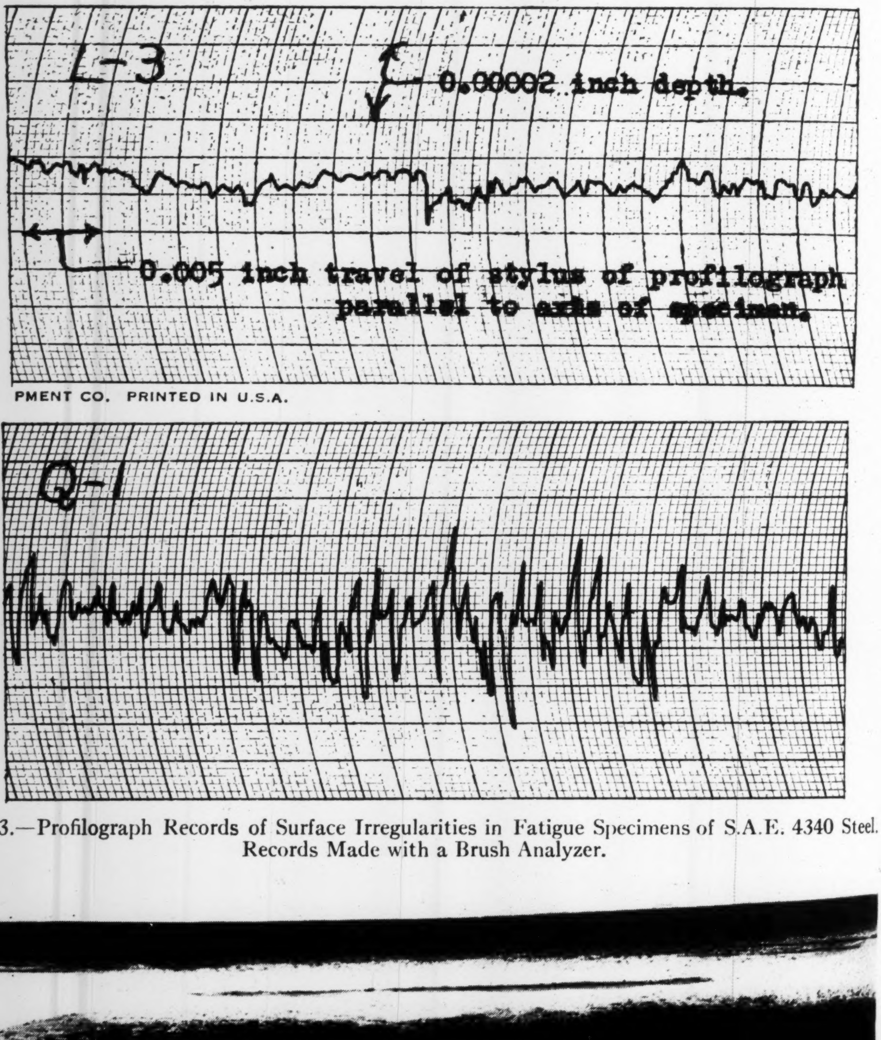

FIG. 3.—Profilograph Records of Surface Irregularities in Fatigue Specimens of S.A.E. 4340 Steel. Records Made with a Brush Analyzer.

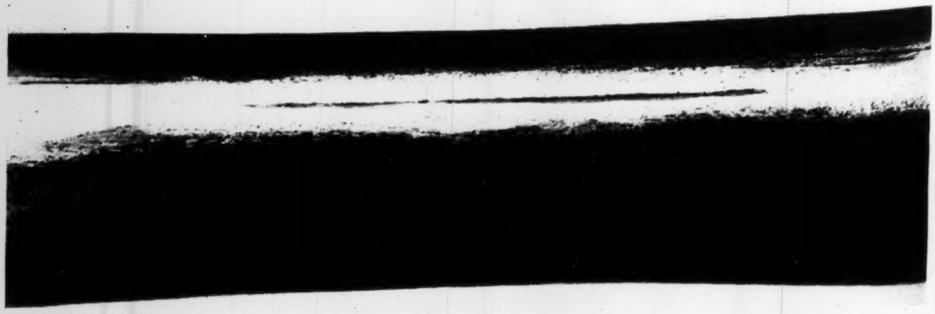

FIG. 4.—Nonmetallic "Stringers" in Fatigue Specimen of Low-Alloy Steel (HT-50) (× 7).

about its axis while a round bar wrapped with emery cloth was held with its axis at right angles to that of the specimen and rotated rapidly. Thus the direction of polishing was very nearly in a longitudinal direction along the specimen. The flat specimens were polished in a longitudinal direction. The final polish of all steel specimens was with No. 00 emery cloth.

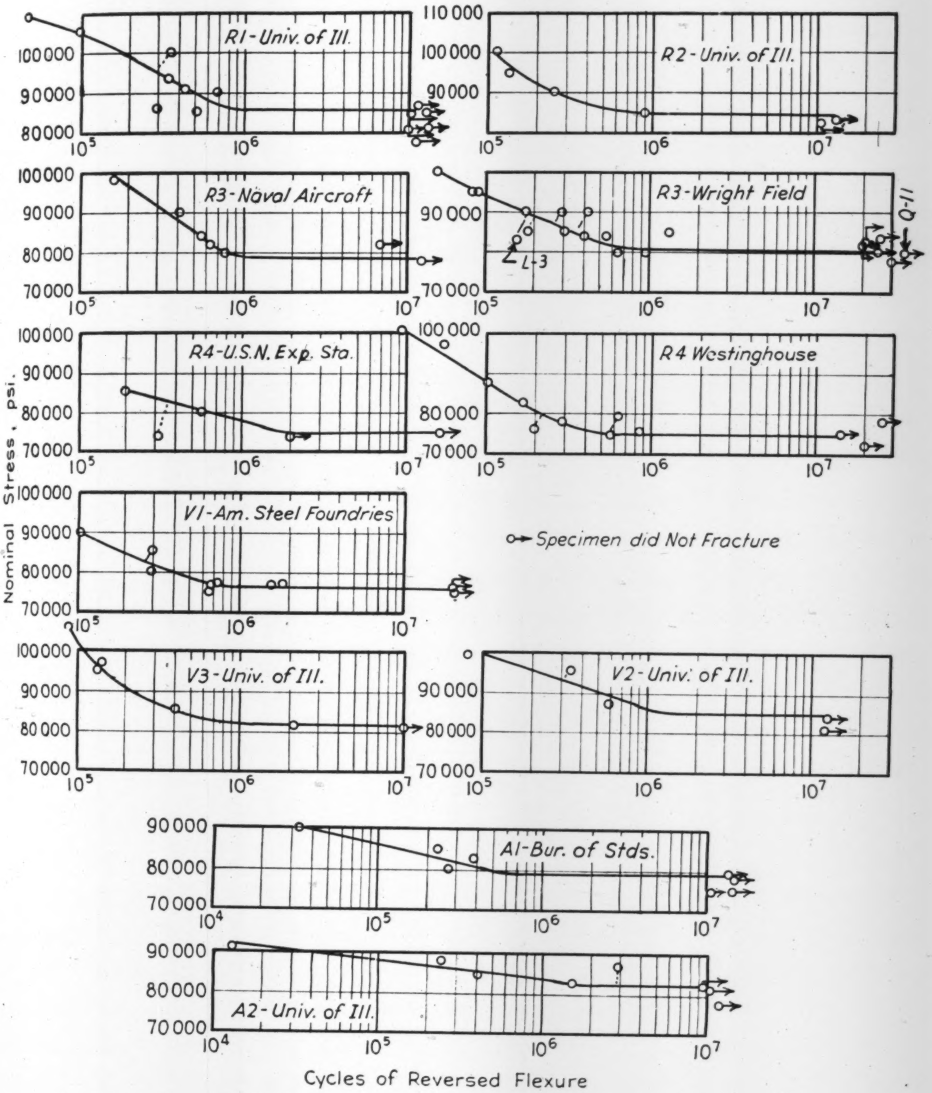

FIG. 5.—*S-N* Diagrams for Heat-Treated Alloy Steel (S.A.E. 4340).

Profilograph Records and Magnaflux Tests:

As some *S-N* diagrams showed considerable "scatter," profilograph records were made at the Timken laboratories on 12 specimens after testing. The two extreme diagrams taken are shown in Fig. 3, Specimen L-3 showing the "smoothest" surface and Specimen Q-1 the "roughest." In Fig. 5, in the *S-N* diagram for the Wright Field tests, the performance of these two specimens is shown. Specimen Q-1, the "roughest" specimen, ran out to 33,000,000 cycles of stress without fracture, just about at the endurance limit as determined by tests of 20 specimens, while L-3, the "smoothest" specimen, failed distinctly below the *S-N* diagram determined from the results of tests of the 20 specimens. Such a result shows no evidence of correlation between the surface as shown by profilograph records and fatigue strength. Examination of the other profilograph records showed similar lack of correlation. This is in harmony with data reported by O. J. Horger of the Timken laboratories.

TABLE II.—ENDURANCE LIMITS (ROUND SPECIMENS).

Metal	Tested at	Testing Machine	Speed, cycles of stress per min.	Nominal Diameter of Specimen in.	Endurance Limit, psi.
Heat-treated alloy steel (SAE 4340)	University of Illinois	R-1 Krouse, rotating cantilever	3 600	0.100	86 000
	University of Illinois	R-2 Boone-Wishart, rotating cantilever	3 600	0.100	84 000
	Naval Aircraft Factory	R-3 R. R. Moore, rotating simple beam	3 450	0.300	79 000
	U. S. Air Service, Wright Field	R. R. Moore, rotating simple beam	3 450	0.300	81 000
	U. S. N. Eng. Expt. Station	R-4 McAdam, rotating cantilever	1 450	0.470	75 000
	Westinghouse Research Lab.	McAdam, rotating cantilever	1 300	0.470	76 000
	American Steel Foundries	V-1 DeForest, magnetic vibratory	8 400	0.312	76 000
	University of Illinois	V-2 Krouse, mechanical vibratory	1 750	0.300	84 500
	University of Illinois	V-3 Moore-Shafer, mechanical vibratory	1 600	0.375	81 000
	National Bureau of Standards	A-1 Haigh, magnetic axial stress	2 400	0.150	79 000
	University of Illinois	A-2 Moore-Krouse, mechanical axial stress	1 100	0.100	82 000
Low-alloy steel, as rolled (HT-50)	University of Illinois	R-1 Krouse, rotating cantilever	3 600	0.125	46 000
	University of Illinois	R-2 Boone-Wishart, rotating cantilever	3 600	0.100	44 000
	Naval Aircraft Factory	R-3 R. R. Moore, rotating simple beam	3 450	0.300	43 000
	U. S. Air Service, Wright Field	R. R. Moore, rotating simple beam	3 450	0.300	45 000
	U. S. N. Eng. Expt. Station	R-4 McAdam, rotating cantilever	1 450	0.470	41 000
	Westinghouse Research Lab.	McAdam, rotating cantilever	1 300	0.470	43 000
	American Steel Foundries	V-1 DeForest, magnetic vibratory	8 400	0.312	48 000
	University of Illinois	V-2 Krouse, mechanical vibratory	1 750	0.300	47 000
	University of Illinois	V-3 Moore-Shafer, mechanical vibratory	1 600	0.400	46 000
	National Bureau of Standards	A-1 Haigh, magnetic axial stress	2 400	0.150	41 000
	University of Illinois	A-2 Moore-Krouse, mechanical axial stress	1 100	0.100	39 000
Duralumin (17S-T)	Aluminum Research Lab.	R-1 Krouse, rotating cantilever	10 000	0.175	15 000[a]
	Aluminum Research Lab.	R-3 R. R. Moore, rotating simple beam	3 500	0.330	15 000[a]
	Aluminum Research Lab.	FC Howell-Howarth, fixed cantilever	3 500	0.408	15 000[a]
	Aluminum Research Lab.	A-3 Templin, mechanical axial stress	2 000	0.200	14 000[a]

[a] Endurance limit for 500 000 000 cycles of stress.

Figure 4 shows results of a magnaflux inspection on a specimen of low-alloy steel (HT-50). Magnaflux examinations of several specimens of HT-50 steel were made in the Materials Laboratory at Wright Field. As in the specimen shown in Fig. 4, evidence was found of longitudinal "stringers," probably nonmetallic inclusions. Specimens of the S.A.E. 4340 steel were sent to Professor DeForest at the Massachusetts Institute of Technology, who reported: "I have run an immersion method Magnaflux inspection of these samples and find them very free from nonmetallic stringers."

COOPERATING LABORATORIES

The polished specimens were shipped to the following laboratories for testing:

Laboratory	Experimenter	Testing Machine
U. S. Air Corps Lab., Wright Field, Dayton, Ohio	J. B. Johnson	R. R. Moore (R3)
U. S. Naval Aircraft Factory, Philadelphia, Pa.	R. R. Moore	R. R. Moore (R3)
U. S. N. Academy, Eng. Exp. Sta., Annapolis, Md.	W. C. Stewart	McAdam (R4)
Westinghouse Research Lab., E. Pittsburgh, Pa.	R. E. Peterson	McAdam (R4)
Nat'l Bureau of Standards, Washington, D. C.	H. S. Rawdon	Haigh (A1)
American Steel Foundries, E. Chicago, Ind.	W. C. Hamilton	Magnetic Vibratory (V1)
University of Illinois, Urbana, Ill.	H. F. Moore	Krouse Cantilever (R1) Boone-Wishart do. (R2) Moore-Krouse (A2) Krouse Vibratory (V2) Moore-Shafer do. (V3)

As noted previously, the specimens of duralumin (17 S-T) were prepared and tested under the direction of R. L. Templin in the Research Laboratories of the Aluminum Company of America, New Kensington, Pa. Tests were made on a Krouse cantilever machine (R1), a R. R. Moore rotating-beam machine (R3), a Howell-Howarth fixed cantilever machine (FC), and a Templin Axial stress machine (A3).

SPEED OF TESTING MACHINES

It would have been desirable to have all tests run at the same speed, but this was not feasible. The speeds used are shown in Table II. There was no evidence of any well-marked "speed effect" in the tests carried out, and this is in harmony with the conclusions of nearly all experimenters who have studied speed effect in fatigue tests, at least for speeds not exceeding the range used in the tests at the various cooperating laboratories with the possible exception of the tests at the American Steel Foundries (8400 cycles of stress per min.) and the Krouse cantilever tests of duralumin (10,000 cycles of stress per min.). In these two series of tests there was no evidence that the endurance limit was raised by the high frequency of loading.[2]

RESULTS OF TESTS

Test Data:

The test data from the various cooperating laboratories was sent to the chairman of the committee, and *S-N* diagrams were plotted by him and endurance limits determined. By having one man plot diagrams and determine endurance limits, it was felt that differences in interpretation of results would be minimized, and the best comparison of results secured. The test data for all tests are on file at the Talbot Laboratory for Materials Testing, University of Illinois.

Figures 5, 6, and 7 show the *S-N* diagrams for the tests of steel specimens. It will be noted that in some diagrams there is shown considerable "scatter" of results but that the line determining the endurance limit seems fairly definite. These tests were carried out under unusually careful conditions, and it seems that "scatter" may be attributed, in part at least, to the variable fatigue strength of metal, even in bars carefully chosen with a view to a maximum degree of uniformity. Adjustment of several of the "scattered" points in the diagrams was attempted by correcting for the varying Brinell and Rockwell C hardness of the bars, but this adjustment did not reduce the "scatter."

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[2] See "Prevention of the Failure of Metals Under Repeated Stress," by the Staff of Battelle Memorial Institute, p. 115, John Wiley and Sons, New York, N. Y. (1941); also *Circular 23*, Engineering Experiment Station, University of Illinois (1941).

Variation of Endurance Limits for Different Machines:

Table II summarizes the endurance limits determined, and Table III tabulates ranges of endurance limit found. It is obvious that to obtain anything like a statistical estimate of probable ranges of values, very many more tests would have to be made than are herein reported, and these ranges of values are reported merely as giving some general idea of ranges obtained in tests made

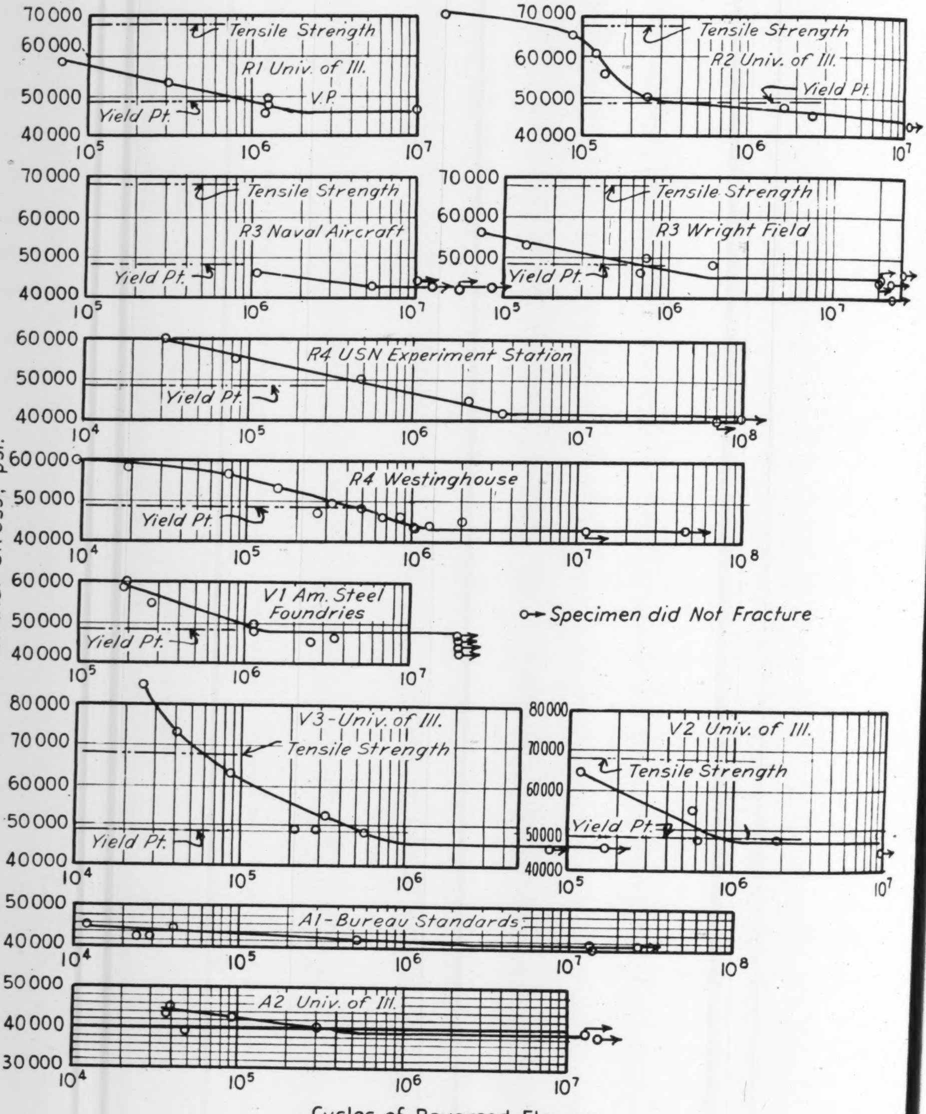

FIG. 6.—*S-N* Diagrams for Low-Alloy Steel (HT-50).

with common types of machines now available and in laboratories experienced in fatigue testing in which the work was carefully done.

TABLE III.—RANGE OF ENDURANCE LIMITS.

Range of Values	Heat-Treated Alloy Steel SAE 4340				Low-Alloy Steel HT-50				Duralumin 17S-T			
	max., psi.	min., psi.	Range		max., psi.	min., psi.	Range		max., psi.	min., psi.	Range	
			psi.	per cent[a]			psi.	per cent[a]			psi.	per cent[a]
NO ADJUSTMENT OF VALUES FOR "SIZE EFFECT."												
All testing machines	86 000	75 000	11 000	13.7	48 000	39 000	9 000	20.7	15 000	14 000	1 000	6.8
Rotating-beam machines	86 000	75 000	11 000	13.7	46 000	41 000	5 000	11.4	15 000	15 000	0	0
Vibratory machines	84 500	76 000	8 500	10.6	48 000	46 000	2 000	4.2	....	....	....	....
Axial-stress machines	82 000	79 000	3 000	3.7	41 000	39 000	2 000	5.0	....	....	....	....[b]
VALUES ADJUSTED TO EQUIVALENT VALUES FOR SPECIMENS 0.3 IN. IN DIAMETER.												
All testing machines	84 500	75 500	9 000	11.3	47 500	38 000	9 500	21.6	15 000	14 000	1 000	6.8
Rotating-beam machines	81 000	79 000	2 000	2.5	45 500	42 500	3 000	6.8	15 000	15 000	0	0
Vibratory machines	84 500	76 200	8 300	10.3	47 500	47 200	300	0.6	....	....	....	....
Axial-stress machines	76 500	75 500	1 000	1.3	40 000	38 000	2 000	5.1	....	....	....	....[b]

[a] This is the ratio of range of stress (in psi.) to mean value of endurance limit expressed in per cent.

[b] Only one axial-stress machine used with duralumin.

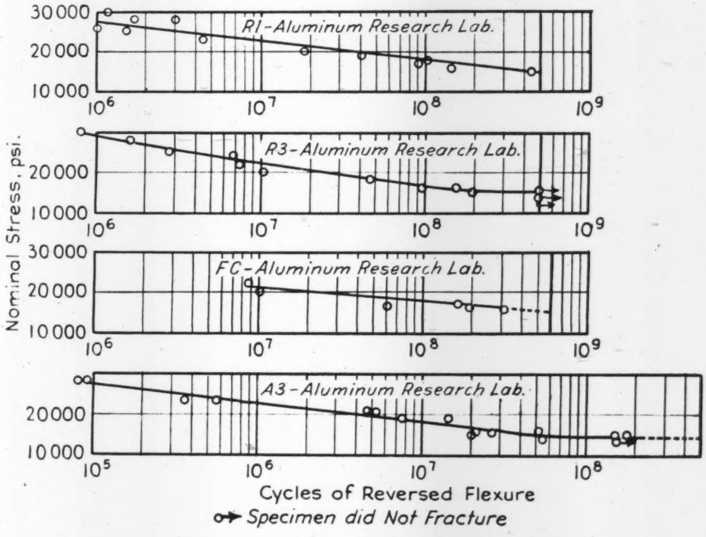

FIG. 7.—*S-N* Diagrams for Duralumin (17 S - T).

The upper portion of Table III shows the ranges for different types of machines and the ranges for all the metals tested, with no adjustment of values. It is seen at once that the highest range of endurance limit determined covers a width of 11,000 psi. This range in pounds per square inch is probably fully as significant as is the range in percentage of mean values, although the percentage variations are given as a matter of interest. It is also noticed that, so far as can be seen, the variation of values for the duralumin seems to be distinctly less both in pounds per square inch and in per cent than the variation for the other materials. Some light is thrown on this variation by Fig. 8 in which endurance limits are plotted as ordinates and values of critical diameter of specimen are used as abscissas. The resulting graphs show distinct evidence of a "size effect" which, over the range tested, is quite pronounced for the heat-treated alloy steel, somewhat less pronounced for the low-alloy steel, and negligible for the duralumin. It must not be assumed that these values can be extrapolated to estimate values for very large specimens. For example, for the rotating-beam tests of the heat-treated alloy steel, extrapolation would show that for a specimen about 3.5 in. in diameter the endurance limit would become zero and the values above that probably negative, an obviously ridiculous result. However, this obvious "size effect" shown in tests of steel is of very great importance in connection with the consideration of fatigue strength of large machine parts. These size effect diagrams shown in Fig. 8 furnish a basis for a further study of variations in endurance limit. The lower part of Table III shows ranges of values of endurance limits adjusted to be equivalent to the endurance limit of a specimen of the same material with a critical diameter of 0.3 in. tested in the same machine. The graphs in Fig. 8 for the rotating-beam endurance limits are used as base line for each metal. For example, the plotted endurance value marked *A* in Fig. 8 (HT-50 steel) is transferred to *A′* on the ordinate line for 0.3 in. diameter. *A′* is the same distance below the base line *MN* as in *A*. Similarly, the plotted endurance limit value *B* is transferred to *B′*. Then *A′Q* gives the adjusted maximum value of endurance limit for the axial stress machines, and *B′Q* represents the adjusted minimum.

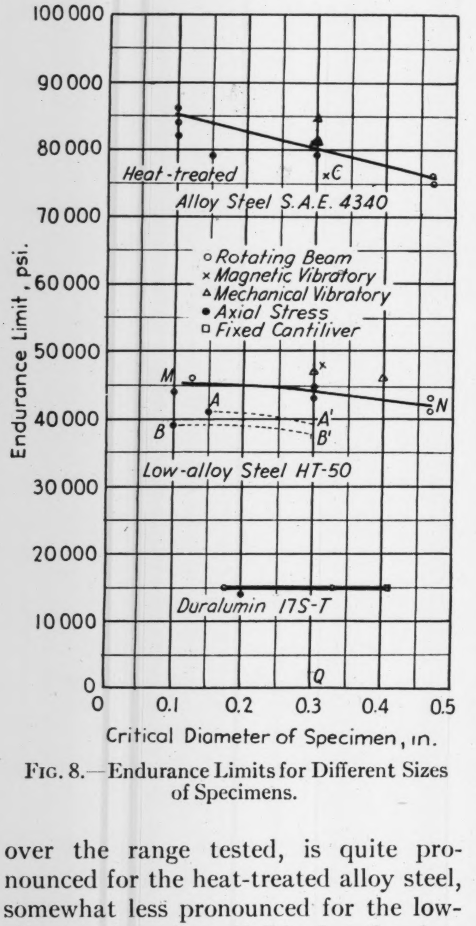

FIG. 8.—Endurance Limits for Different Sizes of Specimens.

It is seen, from Table III, that when results from all testing machines are considered, adjustment for size effect slightly reduces the range of values of endurance limit for the S.A.E. 4340 steel, increases it slightly for the HT-50 steel, and does not change the range of the 17S-T duralumin. The duralumin shows no size effect over the range of diameters tested. However, when the ranges of endurance limits for each different *type* of machine are studied, the range is distinctly reduced by adjustment for "size effect." One exception is found in the case of tests in vibratory machines of specimens of S.A.E. 4340 steel. This exception is largely due to the low value obtained in the tests on the magnetic vibratory machine (see *C*, in Fig. 8). If that value were neglected the variation would be:

Maximum endurance limit.....84 500, psi.
Minimum endurance limit.....81 500, psi., and
Range 3000 psi. or 3.6 per cent of the mean value.

Again noting that the data taken are too few to justify elaborate statistical analysis, the tests do seem to show some evidence that for a given metal, for a given *type* of testing machine and for a given size of specimen, endurance limits determined by different laboratories and different machines of the same type might be expected to vary over a range not greater than 3000 psi. or a variation of, perhaps, 1500 psi. from the mean value by all the laboratories. Again let it be emphasized that this is not presented as a statistical analysis but to give a general idea of the variation which is probable.

The relative low variation in the results of the duralumin may be due partly, perhaps largely, to the fact that tests were made with great care in one laboratory. It seems, however, rather reasonable to assume that this consistency was due, in part at least, to the greater homogeneity of the material. Again it may be emphasized that actual metal is not homogeneous and that repeated stress brings out this lack of homogeneity in a most striking manner.

Comparison of Different Types of Testing Machines:

Making adjustment for size effect as discussed in a previous paragraph, the following mean values were obtained for endurance limits for different types of machines:

Material	Type of Machine	Symbol	Endurance Limit, psi.
Heat-treated Alloy steel SAE 4340	Rotating-beam	(R)	80 000
	Vibratory	(V)	80 600
	Axial stress	(A)	76 000
Low-Alloy Steel HI-50	Rotating-beam	(R)	44 100
	Vibratory	(V)	47 200
	Axial stress	(A)	39 000
Duralumin	Rotating-beam	(R)	15 000
	Vibratory	(V)	....
	Axial stress	(A)	14 000

The ratios of average endurance limits for the different metals and different types of testing machine are as follows:

V/R = 1.008 for 4340 steel; 1.072 for HT-50 steel; no vibratory tests for 17S-T duralumin.
A/R = 0.950 for 4350 steel; 0.884 for HT-50 steel; 0.933 for 17S-T duralumin.

It may be noted that somewhat similar values for the ratios of vibratory machines and rotating-beam machines were obtained by T. J. Dolan on tests of an aluminum alloy. This ratio of vibratory to rotating-beam results would be somewhat larger for the S.A.E. 4340 steel if the low value of the tests on the magnetic vibratory machines were neglected. It may be noted that for the vibratory tests of round specimens only one pair of extreme fibers, top and bottom, are subjected to maximum stress while in the rotating-beam tests *all* the surface fibers are subjected to maximum stress. In the case of the axial-stress tests, the whole cross-section of the specimen is submitted to maximum stress.

Effect of Shape of Specimens:

In carrying out vibratory tests, a few tests were made on flat specimens in place of round specimens, and it was found that flat specimens gave, in general, lower values than did the round specimens. The variation of values is as follows:

	S.A.E 4340 Steel			HT-50 Steel		
	Round	Flat	Ratio	Round	Flat	Ratio
Krouse-Vibratory	84 500	75 000	1.13	47 000	42 000	1.12
Moore-Shafer	81 000	70 000	1.16	45 000	45 000	1.00
			1.14			1.06

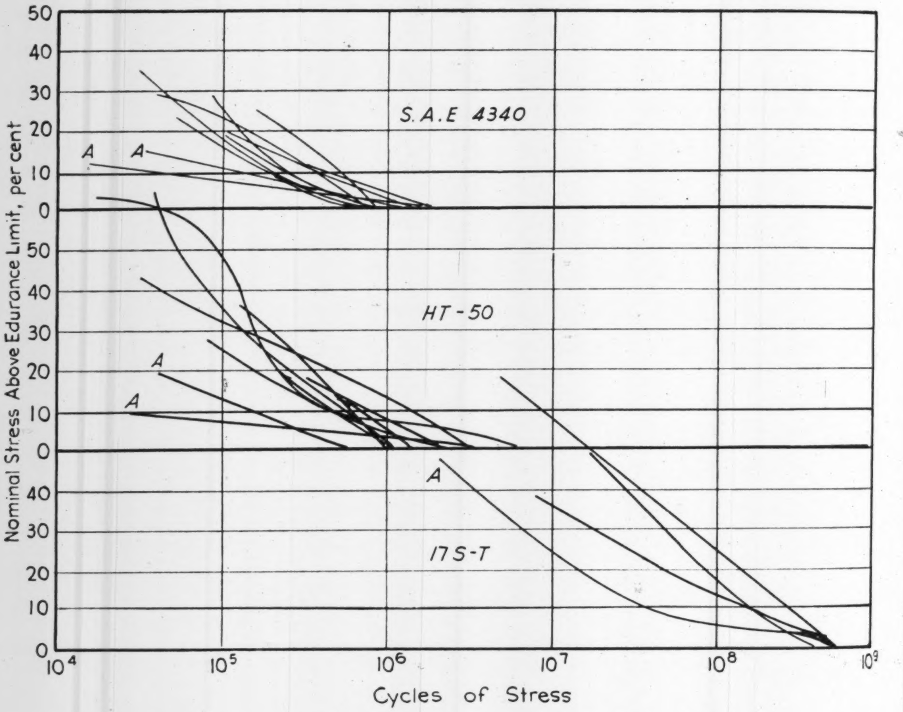

FIG. 9.—Test Results for Specimens above the Endurance Limit.

The flat specimens for S.A.E. 4340 steel were 0.2 by 0.75 in. in cross-section with some specimens 0.3 by 0.5 in. These two sizes of specimens showed practically the same endurance limit. For the HT-50 steel, the flat specimens are all 0.2 by 0.75 in. in cross-section. The diameters of the round specimens are given in Table II. Here again it may be noted that similar results with even more striking differences have been found by T. J. Dolan of the University of Illinois when comparing test results for round and flat specimens of an aluminum alloy, tested in vibratory fatigue testing machines.

This apparent evidence of a shape factor for endurance limit, somewhat resembling the shape factor observed for determinations of yield strength by static flexure tests, is of very considerable interest. Here again it may be noted that for the round specimens only one pair of extreme fibers, top and bottom, were subjected to the vibratory tests, while for the rectangular specimens maximum stress nominally exists across the top and the bottom face. It is also to be noted that the introduction of slight twisting in the flat specimen tests may take place, even when great care is used in placing specimens in the machine.

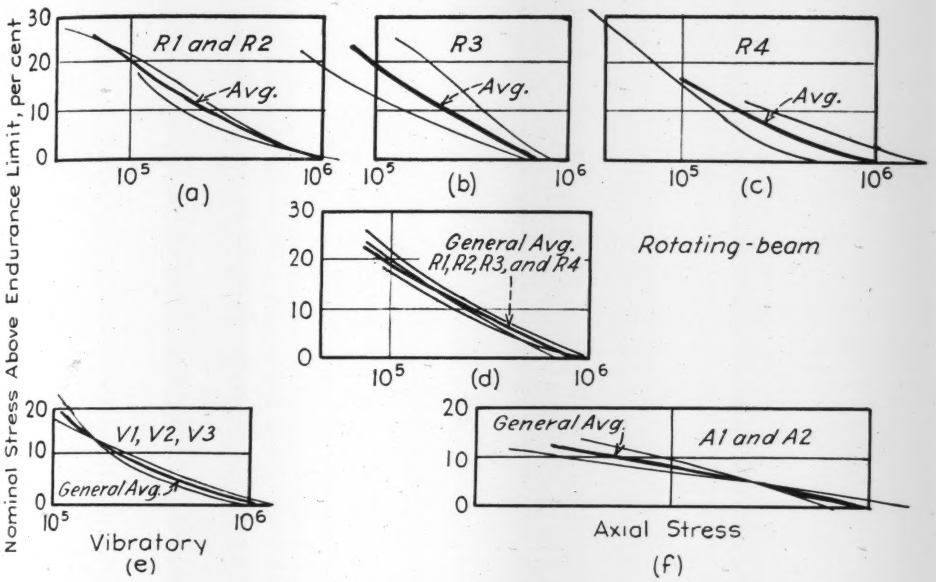

FIG. 10.—Average Test Results above Endurance Limit (S.A.E. 4340 Steel).

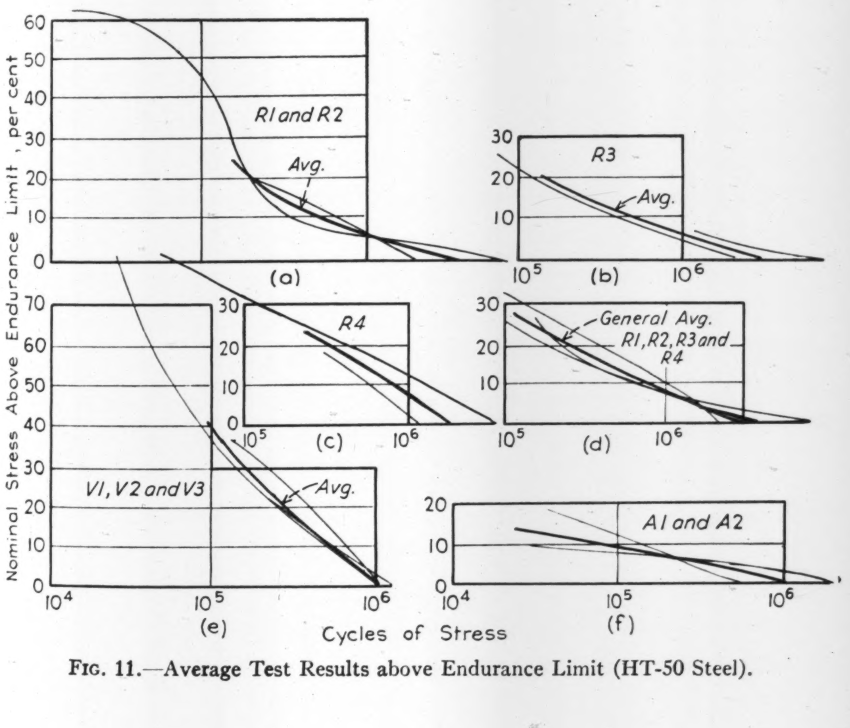

FIG. 11.—Average Test Results above Endurance Limit (HT-50 Steel).

Endurance Above the Endurance Limits:

It has been noted that the "scatter" of test results is greater above the endurance limit than is the scatter of the values of endurance limit determined. This is a quite general experience with experimenters with fatigue of metals. In representing behavior above the endurance limit, the method has been chosen of plotting curves in which ordinates represent stress above endurance limit in per cent of endurance limit, and abscissas represent number of cycles of stress. The use of percentage in place of stress in pounds per square inch above endurance limit was chosen on account of the fact that three different metals were used, and it was thought that a percentage above endurance limit would furnish a better means of comparison than stress above endurance limit. These percentage-cycle curves show the same type of curve as do the *S-N* diagrams in Figs. 5, 6 and 7. However, the scale is different from the scales shown there. Figure 9 shows the percentage-cycle stresses all plotted together, and the result seems to be a confusion of lines with one outstanding fact, namely, that the diagrams for the axial-stress (tension-compression) tests are distinctly flatter than the others. These axial-stress diagrams are marked *A*, Fig. 9. While the data are insufficient for careful statistical analysis, some study of the general tendency of different types of machine and different metals has been made. In Fig. 10 are shown percentage-cycle diagrams for S.A.E. 4340 steel giving comparison of values for different machines which may be regarded as companion machines. For example, Fig. 10 (*a*) shows comparison of diagrams for two rotating-cantilever machines

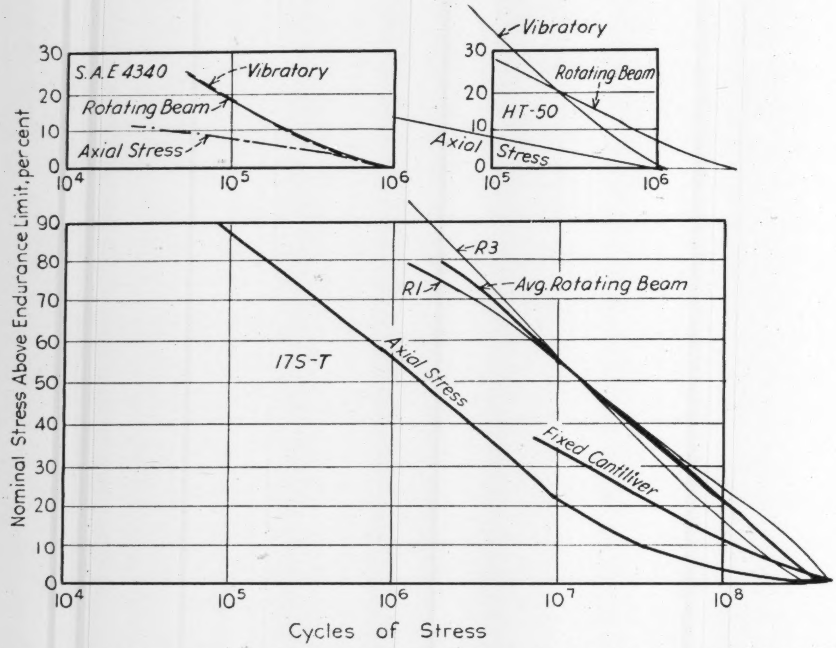

FIG. 12.—Average Test Results above Endurance Limit for Different Types of Testing Machines.

with small specimens. Figure 10 (*b*) shows comparison of values between R. R. Moore machines, Fig. 10 (*c*) shows comparison between two McAdam rotating-cantilever machines. Figure 10 (*d*) shows the three average curves plotted together and a general average curve (heavy line) drawn for all rotating-beam machines. Figure 10 (*e*) shows a similar diagram for the vibratory machines and Fig. 10 (*f*) for the axial-stress machines. Figure 11 shows corresponding graphs for the HT-50 steel.

In Fig. 12 are shown the general averages for rotating-beam machines, vibratory machines, and axial-stress machines for each of the three metals studied. In the case of the duralumin 17S-T, the average of the two rotating-beam machines used (Krouse-cantilever and R. R. Moore) is shown. Taking all these values into consideration, it is obvious that the scatter between individual specimens used for determining an *S-N* diagram, and the variation between the percentage-cycle or the *S-N* diagrams above the endurance limit for companion machines of the same type, is greater than the variation in the average result obtained for all machines of one type. In Fig. 12 it is noted that, for the steels, the vibratory and rotating-beam type of machines seem to give average curves lying quite closely together, especially for the S.A.E. 4340 steel. In the curve for the 17S-T duralumin, the fixed-cantilever type of machine seems slightly flatter than that for the rotating-beam. In all cases the diagrams above the endurance limit are flatter for the axial-stress machines than for the rotating-beam or vibratory type machines. Figure 13 shows the general average diagram for the rotating-beam machines for the three metals. Evidently the steel reached the endurance limit at a much smaller number of cycles of stress than the 17S-T specimens.

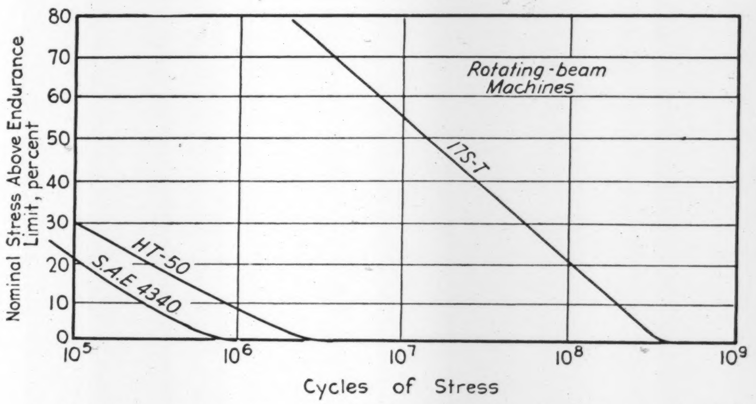

FIG. 13.—Average Test Results above Endurance Limit for Rotating-Beam Tests of Different Metals.

In tests of HT-50 steel in machine R2 and V3 (see Fig. 6), it is noted that some tests have been run not only above the yield point of the steel, but in these two *S-N* diagrams the maximum nominal stress was distinctly above the tensile strength of the steel. This, of course, merely shows that the nominal stress above the yield point is very much higher than the actual stress. In fact, it is doubtful whether the actual stress rises much above the yield point.

Tests on machine R1 in Fig. 5 and R2 in Fig. 6 show a distinct reversal of curvature, but the significance of this is uncertain since tests on machines V3 in Fig. 6 do not show such reversal.

ACKNOWLEDGMENTS

Acknowledgments are made to the Timken Roller Bearing Co. and to O. Horger for furnishing the heat-treated alloy steel S.A.E. 4340, for profilograph tests and for micrographs of the S.A.E. 4340 steel; to the American Rolling Mill Co. and to R. L. Kenyon for furnishing the low-alloy steel HT-50; to the Aluminum Company of America for permission to use the data on tests of duralumin 17S-T; to the U. S. Air Corps laboratories at Wright Field and to J. B. Johnson for magnaflux tests and micrographs, to A. V. DeForest of the Massachusetts Institute of Technology for magnaflux tests, and to the laboratories and the men who carried out the testing work for this investigation.

DISCUSSION

MR. J. M. LESSELLS[1] (*presented in written form*).—The report of the Research Committee on Fatigue of Metals is of considerable interest since it brings much new data on testing machines together for the first time. Some of the plotted results show considerable "scatter" and some comment on this phenomenon in the light of experience with the R.R. Moore type of machine may be of interest.

From the nature of fatigue tests it is generally accepted that there must be scatter in the results. It appears that this scatter may be caused by (1) condition of material as regards cleanliness or residual stress, (2) condition of the surface of the test specimens and of machine operation. Provided the steel is relatively free from nonmetallic inclusions and residual stress and care is observed in the subsequent testing it is our opinion that the amount of scatter can be held to a minimum. In some cases where these precautions have been taken, remarkable regularity in the fatigue data has been obtained.

In the fatigue testing work which has been done at the Massachusetts Institute of Technology during recent years, it has been observed that steels purchased on the basis of grain-size control have given little or no scatter. This has been particularly true when these materials have been tested in the annealed condition and presumably with little or negligible residual stress present to cause variation. Cases of carefully controlled heat-treated steels have however also shown little or no scatter.

The A.S.T.M. instructions as to preparation of test specimens are good and should be followed. The finishing should be such that the scratches, if any, are in a longitudinal direction rather than circumferential. Buffing should be done with care, and hand polishing is preferred. Small diameter specimens, such as can be tested from S.A.E. 4340 steel on an R. R. Moore type of machine, can be easily bent by the buffing operation.

Precautions should be taken to ensure that all specimens run smoothly. This can be done by taking the weight hanger off and making sure that the bearings operate smoothly when the specimen is running without load. This is important since it is relatively easy to cause misalignment in setting up. The ends of the specimen must fit the gage properly and load should be applied after the machine is running.

Dead weight loading seems preferable to spring loading.

The M.I.T. machine operates at 1750 rpm., which is a relatively low speed with absence of vibration. Higher speeds may give rise to vibration effects. The practice of testing a specimen which does not run perfectly true at zero load is in our opinion bad and may be worse for the higher speed machines.

The scope of the M.I.T. fatigue work has covered such materials as Armco iron, S.A.E. 1020, S.A.E. 1045, and S.A.E. 4340 steels. All of these materials have shown very little scatter although as experienced in other laboratories, many materials have been tested which have shown considerable scatter

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[1] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

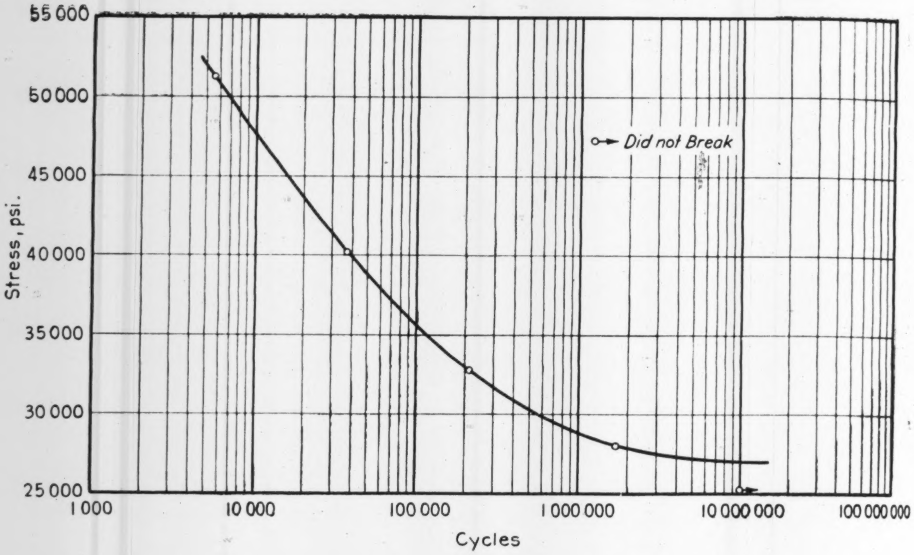

FIG. 1.—Armco Iron.

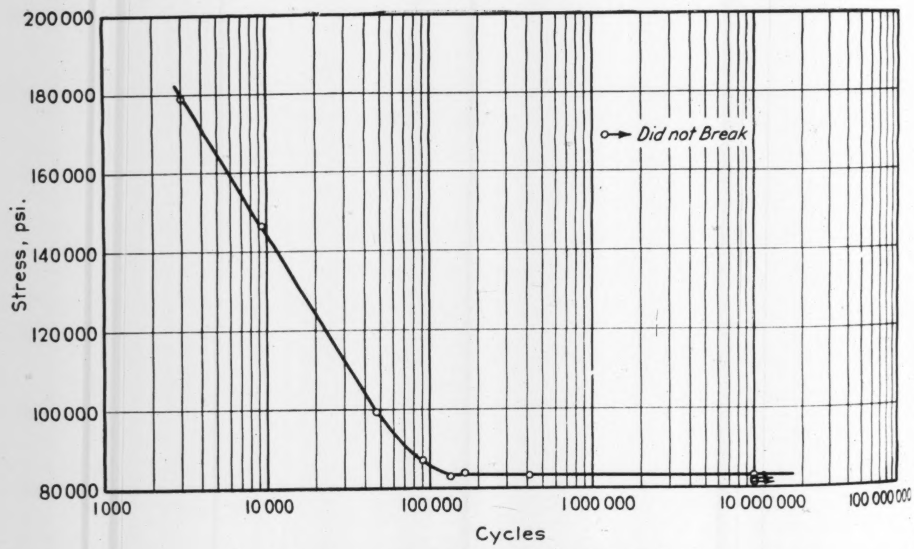

FIG. 2.—S.A.E. 4340 Steel (Heat Treated).

but these presumably possessed some of the defects cited above.

For the purpose of illustration the results for Armco iron, which is a relatively soft material, and those for an oil-quenched and tempered S.A.E. 4340 steel, which is a relatively hard material, are given in Figs. 1 and 2.

MR. H. F. MOORE.[2]—Commenting on Mr. Lessells' remarks concerning "scatter," it may be noted that in the tests reported by the Fatigue of Metals Committee as made on R. R. Moore machines, laboratories were chosen in which it was known that great care was taken to have the specimens run smoothly with a minimum of vibration. It may be further noted that the tests of heat-treated alloy-steel specimens showed greater tendency to exhibit "scatter" than was exhibited in the tests of low-alloy steel specimens or in tests of duralumin specimens. It is also worthy of note that, with the exception of one series of tests of small cantilever specimens, the rotating-cantilever tests, the vibratory tests and the reversed-axial-stress tests (push-pull tests) showed no greater "scatter" than did the tests made in R. R. Moore machines.

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[2] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

REPORT OF JOINT RESEARCH COMMITTEE

ON

EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

Progress Report to the Sponsor Societies

Meetings:

During the past year, the Joint Research Committee on Effect of Temperature on the Properties of Metals under the sponsorship of the American Society of Mechanical Engineers and the American Society for Testing Materials, held one meeting on November 1 and 2, 1940, in Columbus, Ohio. A second meeting of the committee will be held in June, 1941, in Chicago, Ill., prior to the presentation of this report at the annual meeting of the A.S.T.M. A number of meetings of project committees have been held during the year.

The committee sponsored a technical session on December 5, 1940, in New York City, at the annual meeting of the A.S.M.E., at which two technical papers[1,2] and one progress report[3] were presented.

Finances:

There appears in Table I a summary of the administration of funds during the yearly period of May 1, 1940, to April 30, 1941.

Expenditures have been authorized and payments made to date on several research projects, as follows:

	Maximum Expenditure Authorized	Payments Made
Project No. 10—Tubular Members Subject to Internal Pressures	$3 500.00	$3 500.00
Project No. 18—Effect of Variables on the High-Temperature Properties of Metals (Part 3)	4 000.00	1 892.81
Project No. 23—Studies on Non-Ferrous Alloys	500.00	none
Project No. 25—Comparison of Short-Time Methods	1 000.00	500.00

Organization and Membership:

The committee records with deep regret the passing away on February 18, 1941, of Paul E. McKinney, one of its most active members. Mr. McKinney joined the committee as a member in June, 1931. He had always taken a most active interest in the work, serving as chairman of several of the project committees. His knowledge on the metallurgy and production of steel was particularly valuable to the work of the committee.

One new member has been added to the membership, Claude L. Clark, of the Steel and Tube Division of the Timken Roller Bearing Co. Mr. Clark has long been actively engaged in the work of the committee as a cooperator and advisor, and has handled research activities for the committee on several occasions.

No other changes in membership nor in organization have taken place during the year.

Research Projects:

There follows a summary of progress made in the various research projects during the year ending June, 1941.

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[1] A. W. Wheeler, "A High-Temperature Bolting Material," Preprint A.S.M.E., December, 1940.

[2] A. E. White and Sabin Crocker, "Effect of Grain Size and Structure on Carbon-Molybdenum Steel Pipe," Preprint A.S.M.E., December, 1940.

[3] F. H. Norton, "Progress Report on Tubular Creep Tests." C. R. Soderberg, "Interpretation of Creep Tests on Tubes," Preprints, A.S.M.E., December, 1940.

Project No. 10 on Tubular Members Subjected to Internal Pressures (H. J. Kerr, chairman).—Reference has already been made to the presentation of a progress report by F. H. Norton[3] and a note on interpretation by C. R. Soderberg[3] at the technical session sponsored by the committee during the annual meeting of the A.S.M.E. in December, 1940. It is expected that Project No. 10 will complete the work assigned so that a final report can be made at the 1941 annual meeting of the A.S.M.E.

Project No. 13 on Properties of Metals at Low Temperature (F. B. Foley, chairman).—Following the Round-Table Discussion on Effect of Subatmospheric Temperatures on the Properties of Metals[4] at the June, 1939, annual meeting of the A.S.T.M., the project committee prepared and circulated special forms to facilitate the gathering of data on the effect of low temperatures on the properties of metals. Approximately 60 possible sources of information were contacted. The considerable amount of data collected was turned over to Bat-

TABLE I.—SUMMARY OF RECEIPTS AND EXPENDITURES OF COMMITTEE FUNDS.

Balance as of April 30, 1940, shown in 1940 report			$10 149.77
	RECEIPTS		
1940			
May	1939-40 Solicitation for funds	$200.00	
June	1939-40 Solicitation for funds	500.00	
July	1939-40 Solicitation for funds	575.00	
	The Engineering Foundation	500.00	
August	1939-40 Solicitation for funds	4 175.00	
September	Interest on funds	30.75	
November	1939-40 Solicitation for funds	150.00	
December	1939-40 Solicitation for funds	50.00	
1941			
January	1939-40 Solicitation for funds	75.00	
April	1939-40 Solicitation for funds	1 000.00	
	Total Receipts		7 255.75
			$17 405.52
	EXPENDITURES		
1940			
May	Massachusetts Inst. of Technology (Project No. 10)	$700.00	
	Battelle Memorial Institute (Project No. 18)	343.49	
June	Paper and Postage for Project No. 13	6.73	
September	Massachusetts Inst. of Technology (Project No. 10)	700.00	
	A.S.T.M. (reprints of Round-Table Discussion (Project No. 13))	4.20	
October	Finance Committee Expenses	21.25	
November	A.S.M.E. (mimeograph paper for Progress Report of Project No. 10)	6.12	
December	Battelle Memorial Institute (Project No. 18)	314.43	
1941			
January	Ogilvee Press (letterheads)	7.91	
	National Process Co. (300 copies of Project No. 10 Report)	14.35	
	St. John Associates (300 copies of Project No. 10 Report)	43.89	
February	Battelle Memorial Institute (Project No. 18)	205.69	
	Massachusetts Inst. of Technology (Project No. 10)	700.00	
April	Battelle Memorial Institute (Project No. 18)	1 372.69	
	Battelle Memorial Institute (Project No. 13)	500.00	
	University of Michigan (Project No. 25)	500.00	
	Bossow's (flowers, P. E. McKinney)	7.75	
	Total Expenditures		5 448.50
Balance on hand, April 30, 1941			$11 957.02

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[4] F. B. Foley, "Résumé of Round-Table Discussion on Effect of Subatmospheric Temperatures on the Properties of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 642.

telle Memorial Institute in November, 1940, for critical review under the direction of H. W. Gillett. There has been considerable urge from those engaged in national defense matters to make this review available as quickly as possible. Because of this, the review and compilation of data have been prepared without awaiting formal committee action and has been issued by the A.S.T.M. in a separate publication, entitled "Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures," prepared by H. W. Gillett, August, 1941.

Project No. 16 on Relaxation (E. L. Robinson, chairman).—Progress has been made in the arrangements to prepare a final report, or sponsor a session on relaxation.

Project No. 18 on Effect of Variables on the High-Temperature Properties of Metals (H. W. Gillett, chairman).—The 1940 report of the committee includes a report[5] on this project showing the effects of various heat treatments on the creep resistance of pure iron, rimmed steels, silicon-killed, aluminum-killed, and silicon-aluminum-killed steels. The hypothesis was advanced that it was the absence or presence of inclusions of various types and compositions whose size, mode, and degree of dispersion changed with the various heat treatments which produced such large differences in their creep resistance.

The project has been continued during the past year with a further study of some of the steels already tested, and new experiments on a rimmed steel to which 0.58 per cent molybdenum was added, on a fine-grained nonaging steel, on some weld metal with and without 0.5 per cent molybdenum, and on the British steel "E" noted for its inferior creep properties and nonresponse to heat treatment.

Addition of 0.58 per cent molybdenum to a rimmed steel eliminated the poor creep resistance usually obtained with rimmed steel. The creep resistance was little affected by heat treatment and was about equal to that shown by killed steels of similar molybdenum content.

The usual large improvement in creep resistance obtained from normalizing after coarsening of aluminum-killed steels did not result in the case of the fine-grained nonaging steel with higher aluminum addition. Experiments on the effect of more rapid cooling are now in progress.

Tests are now in progress on the effect of heat treatments on the weld metal and the British steel "E."

The work will be reported in detail to the Joint Committee meeting in June, but a progress report to the sponsor societies will not be submitted at this time since the work is incomplete.

Project No. 25 on Comparison of Short-Time Test Methods (J. S. Worth, chairman).—This project is now active. Thirteen laboratories are cooperating in the work. An agreement has been entered into with the University of Michigan to carry out some of the tests, to act as the clearing house for the many reports from cooperators, and to prepare the final report. The project involves the comparison of results from nine shorter-time test methods with those from long-time creep tests on two steels, the 0.35 per cent carbon steel K-20, and a carbon-molybdenum steel K-22.

Project No. 26 on Test Methods (J. W. Bolton, chairman).—This project committee is charged with the review of the two tentative test methods under the jurisdiction of the Joint Research Committee, and with the study and development of other test methods to determine high temperature properties. As a result of the work of this project committee, recommendations are made below relative to the revision and adoption as a standard A.S.T.M. recommended practice of the Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 - 38 T).[6]

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[5] "Study of Effects of Variables on the Creep Resistance of Steels," (Project No. 18), by H. C. Cross and J. G. Lowther, Appendix I of Report of Joint Research Committee, *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 125 (1940).

The committee recommends that the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 - 37 T)[6] be continued as tentative for another year.

ADOPTION AS STANDARD OF TENTATIVE STANDARD

The committee recommends that the Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 - 38 T)[6] be approved, with the following revisions, for reference to letter ballot of the Society for adoption as standard:

Title.—Change to read as follows:

Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials.

Section 1 (a).—Change the words "this method" to read "this recommended practice."

Section 2 (a).—Change from its present form to read as follows:

(*a*) Inherently the creep test is a long-time test and not convenient for acceptance purposes. The primary use of creep tests is to provide basic data for the choice of safe working stresses for applications in which allowable deformation within the service life of the structure is the criterion of failure.

Section 6 (c).—Add a new Paragraph (*c*) to read as follows, relettering the present paragraph as Paragraph (*d*):

(*c*) Care should be taken to obtain reliable and accurate master temperature standards and to have these checked at suitable intervals. When thermocouples are used in the test, these should be checked by suitable means both before and after long-time tests.

Section 9 (d).—Add a new Paragraph (*d*) to read as follows:

(*d*) After unloading, a certain amount of prolonged plastic recovery occurs, in addition to the elastic recovery. This plastic recovery should be recorded.

Section 11 (c).—Add the following new sentence at the end of this paragraph:

Although the plotting of data on semilog charts often results in a straight line relationship, extrapolation of such lines has been found to be unjustified.

Appendix II.—In Section I add a new heading for "Ferrous Alloys" and under this heading add columns for the inclusion of data on chemical composition, austenitic grain size established, grain size in specimen as tested, and normality.

Also add a new heading to cover "Non-Ferrous Alloys" to read as follows:

NON-FERROUS ALLOYS
Chemical Composition:
Cu Sn Zn Pb Ni Fe Al
P Si Mn Others

In Section II change the listing of items for the Type Furnace under Manufacturing Data to read as follows by the addition of the italicized words and the omission of those in brackets:

Type Furnace
Acid or Basic:
[Electric] *Direct* Arc:
Indirect Arc:
Induction:
Open Hearth:
Open Flame:
Crucible:

In Section III change the title to read "Properties of Specimens After Tests." Add a new column with the heading "Temperature" between the present third and fourth columns. Change the heading of the fifth column from "Yield Point" to read "Yield Strength (Offset, 0.2 per cent)."

This recommendation has been submitted to letter ballot of the committee which consists of 22 members; 22 members returned their ballots, of whom 21 have voted affirmatively, 0 negatively, and 1 member has marked his ballot "not voting."

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[6] 1939 Book of A.S.T.M. Standards, Part I, pp. 1261, 1254.

This report has been submitted to letter ballot of the committee which consists of 22 members; 22 members have returned their ballots, of whom 20 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the Joint Research Committee,

N. L. MOCHEL,
Chairman.
H. J. KERR,
Vice-Chairman.

J. W. BOLTON,
Secretary.

REPORT OF COMMITTEE B-1
ON
COPPER AND COPPER-ALLOY WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors held two meetings during the year: one in Atlantic City, N. J., on June 27, 1940, and one in New York, N. Y., on April 14, 1941.

Several additions and changes in the membership of the committee have been made during the year, but the total membership of 41 remains the same with 18 producer, 15 consumer, and 8 general interest members. In addition, 12 other individuals through their company affiliations augment this membership by participating in the subcommittee work of this committee.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II (P. H. Grogan, chairman) *and Subcommittee VII* (R. S. Pratt, chairman) have continued their studies on the effect of speed of testing and are correlating the information obtained with the findings of Committee E-1 on Methods of Testing.

Subcommittee IV (J. R. Becker, chairman) is continuing the study of definitions and requirements for the shapes of edges and corners of rectangular and square copper wire.

Subcommittee V (P. H. Grogan, chairman) is continuing the work on a tentative revision of the Standard Specifications for Hot-Rolled Copper Rods for Electrical Purposes (B 49 - 39), to provide a proper twist test and a definition as to what constitutes major cracks in rods.

Subcommittee VIII (W. H. Bassett, Jr., chairman) is continuing the preparation of Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors. A preliminary draft of these specifications was published in the preprint copy of the 1940 report of Committee B-1. At the June, 1940, meeting it was decided that these specifications should be further developed before submission to the Society. This has been accomplished and the proposed tentative specifications which have been submitted for letter ballot are appended hereto.[1] These specifications make desirable a restriction of the scope of the Standard Specifications for Bare, Stranded Copper Cable, Hard, Medium-Hard, or Soft (B 8 - 39)[2] to concentric-lay-stranded construction, and the subcommittee is accordingly recommending such a revision as given later in this report.

Subcommittee X (C. S. Gordon, chairman) has prepared specifications for lead and lead-alloy coated copper wire of hard-drawn, medium-hard-drawn, and annealed grades which have been circulated among members of this subcommittee. Interest in these specifications is rather keen at the present time due to the scarcity of tin. These specifications will shortly be sent to all members of Committee B-1 for comments and suggestions with the hope that a draft suitable for presentation to the Society as tentative can be formulated within the coming year.

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[1] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 369.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 584.

Subcommittee XI (R. S. Pratt, chairman) is investigating the needs for specifications for special quality copper wire. The chairman of this subcommittee has reported that a subcommittee of Committee B-2 on Non-Ferrous Metals and Alloys has been formed to prepare specifications for oxygen-free copper for electrical purposes and that Subcommittee XI will postpone its work until the subcommittee of Committee B-2 has had the opportunity to complete its specifications.

Subcommittee XII (Alfred Bellis, chairman) has prepared a draft of specifications for tinned-hard or medium-hard copper wire for electrical conductors and has received some comments from members of the subcommittee. Questions relating to tensile strength and elongation values and gage lengths to be used during testing predominate. It has been tentatively decided to consider for the present, wire which has been tinned after the drawing processes have been completed, leaving for future consideration wire drawn after tinning. It has also been decided to prescribe tension tests on the basis of specimens with 10-in. gage lengths for the present, leaving the 60-in. gage lengths for future consideration. It has also been decided to restrict the scope of these specifications to wire not smaller than 0.040 in. in diameter. Tentatively tensile strength values approximately 10 per cent lower than those prescribed for uncoated wire of corresponding tempers are being considered with an additional 5 per cent allowance for stranding.

Subcommittee XIII (W. H. Bassett, Jr., chairman) is considering the various methods of making bend tests and the types of testing equipment available. The investigation has not been completed and no recommendations have been made.

Subcommittee XIV (Alfred Bellis, chairman) was formed to study the use of wire bar specifications in wire specifications and has reported that there was very little response to the questionnaire sent out to Committee B-1 requesting opinions on the adequacy of the Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 27), and for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 27) for use in the wire specifications. It has been decided to discontinue this subcommittee and designate Alfred Bellis and G. H. Frank to work with a similar group from Committee B-2 on Non-Ferrous Metals and Alloys, since Specifications B 4 and B 5 are under the jurisdiction of Committee B-2.

This joint committee has recommended to Committee B-2 that they consider changing the resistivity requirements for electrolytic copper wire bars in Specifications B 5 and the resistivity requirements for low resistance lake copper for lake copper wire bars in Specifications B 4 from the present maximum of 0.15436 ohm (meter, gram) to the new value of 0.15328 ohm (meter, gram). This lowering of the maximum resistivity requirement will raise the conductivity requirement from 99.3 per cent to an equivalent requirement of 100 per cent conductivity. Committee B-1 is proposing the lowering of the resistivity requirements of the Standard Specifications for Soft or Annealed Copper Wire (B 3 – 39) and the Standard Specifications for Hot-Rolled Copper Rods for Electrical Purposes (B 49 – 39) to increase the equivalent conductivity requirement from 98.1 per cent to a minimum requirement of 100 per cent conductivity. This has been submitted to letter ballot of this committee for approval on the basis that Committee B-2 changes the resistivity requirements in the wire bar specifications to a compensating figure. It is planned to compile a complete list of the various resistivity and corresponding conductivity requirements of the copper products covered in the A.S.T.M. specifications under the jurisdiction of Committee B-1, Committee B-2, and Committee B-5 on Copper and Copper Alloys and submit it to the members of Committee B-1 for study.

Subcommittee XV (G. E. Rolston, chairman) is studying the dimensional tolerances prescribed in the various specifications prepared under the jurisdiction of Committee B-1. The purpose of this study is to remove as far as possible the inconsistencies in dimensional tolerances of similar specifications. The immediate study is to include the Standard Specifications for Bare, Stranded Copper Cable, Hard, Medium-Hard, or Soft (B 8 – 39)[2] and the proposed Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors.[1] A comparison is to be made of these requirements with industry requirements as given in the Insulated Power Cable Engineers Association Specifications for Varnished Cambric Insulated Cables, the Underwriters' Laboratory Standards for Rubber-Covered Wire, the Association of Edison Illuminating Companies Specifications for Paper Insulated, Lead-Covered Cable, the A.S.T.M. Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 40 T), and other industry specifications covering stranded wire. Certain revisions relative to dimensional tolerances in the Standard Specifications for Hard-Drawn Copper Wire (B 1 – 40) and for Medium-Hard-Drawn Copper Wire (B 2 – 40) were adopted by the Society last year.

This subcommittee is continuing its work on the other assignments and will endeavor to make recommendations for area tolerances which are acceptable to the industry and which will make the specifications more generally applicable.

Subcommittee XVI (R. S. Pratt, chairman) is studying certain dimensional changes in the Standard Specifications for Bronze Trolley Wire (B 9 – 39) and for Copper Trolley Wire (B 47 – 39) in order to clarify Fig. 1 of these specifications showing the standard sections of grooved trolley wire, and in order to indicate that certain dimensional tolerances are for the information of the die makers and are not intended as the dimensions of the finished product. In studying this matter this subcommittee has found it desirable to provide a micrometer gage for checking the dimensions of the finished wires. A design for this gage has been prepared and discussed, but it has been found necessary to review the design further before it will be acceptable to all members.

REVISIONS OF STANDARDS

The committee recommends for immediate adoption revisions in the Standard Specifications for Bare, Stranded Copper Cable, Hard, Medium-Hard, or Soft (B 8 – 39) as indicated below, and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society. These revisions are necessitated by the preparation of new proposed Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors[1] which restrict the scope of Specifications B 8:

Title.—Change to read as follows by the addition of the italicized words and the omission of the word in brackets:

> Specifications for [Bare,] *Concentric-Lay-*Stranded Copper Cable, Hard, Medium-Hard, or Soft.

TABLE I.—DIAMETERS, AREAS, AND WEIGHTS OF CONCENTRIC-LAY-STRANDED COPPER CABLES,

Size of Cable, cir. mils or A.w.g. numbers	Nominal Cable Diameter, in.[a]			Area, sq in.	Weight	
	Class AA	Class A	Class B		per 1000 ft-lb.	per mile, lb.
*5 000 000 cir. mils	....	2.580	2.581	3.927	15 890	83 910
4 500 000 cir. mils	....	2.448	2.448	3.534	14 300	75 520
4 000 000 cir. mils	....	2.307	2.309	3.142	12 590	66 490
3 500 000 cir. mils	....	2.158	2.159	2.749	11 020	58 180
*3 000 000 cir. mils	....	1.998	1.998	2.356	9 353	49 390
*2 500 000 cir. mils	....	1.823	1.824	1.963	7 794	41 150
*2 000 000 cir. mils	....	1.630	1.632	1.571	6 175	32 600
1 900 000 cir. mils	....	1.590	1.590	1.492	5 866	30 970
1 800 000 cir. mils	....	1.547	1.548	1.414	5 558	29 340
*1 750 000 cir. mils	....	1.526	1.526	1.374	5 403	28 530
1 700 000 cir. mils	....	1.504	1.504	1.335	5 249	27 710
1 600 000 cir. mils	....	1.459	1.459	1.257	4 940	26 080
*1 500 000 cir. mils	....	1.411	1.412	1.178	4 631	24 450
1 400 000 cir. mils	....	1.364	1.364	1.100	4 323	22 820
1 300 000 cir. mils	....	1.314	1.315	1.021	4 014	21 190
*1 250 000 cir. mils	....	1.288	1.289	0.9817	3 859	20 380
1 200 000 cir. mils	....	1.263	1.263	0.9425	3 705	19 560
1 100 000 cir. mils	....	1.209	1.209	0.8639	3 396	17 930
*1 000 000 cir. mils	1.151	1.152	1.152	0.7854	3 088	16 300
900 000 cir. mils	1.092	1.094	1.094	0.7069	2 779	14 670
*800 000 cir. mils	1.029	1.031	1.031	0.6283	2 470	13 040
*750 000 cir. mils	0.997	0.998	0.998	0.5890	2 316	12 230
*700 000 cir. mils	0.963	0.964	0.964	0.5498	2 161	11 410
650 000 cir. mils	0.928	0.929	0.929	0.5105	2 007	10 600
*600 000 cir. mils	0.891	0.891	0.893	0.4712	1 853	9 781
550 000 cir. mils	0.853	0.853	0.855	0.4320	1 698	8 966
*500 000 cir. mils	0.811	0.813	0.813	0.3927	1 544	8 151
450 000 cir. mils	0.770	0.772	0.772	0.3534	1 389	7 336
*400 000 cir. mils	0.726	0.726	0.728	0.3142	1 235	6 521
*350 000 cir. mils	0.710	0.679	0.681	0.2749	1 081	5 706
*300 000 cir. mils	0.657	0.629	0.630	0.2356	926.3	4 891
*250 000 cir. mils	0.600	0.574	0.575	0.1963	771.9	4 076
*No. 0000	0.522	0.552	0.528	0.1662	653.3	3 450
*No. 000	0.464	0.492	0.470	0.1318	518.1	2 736
*No. 00	0.414	0.414	0.419	0.1045	410.9	2 169
*No. 0	0.368	0.368	0.373	0.08289	325.8	1 720
No. 1, 3 wire	0.360	....	....	0.06573	255.9	1 351
*No. 1	....	0.328	0.332	0.06573	258.4	1 364
No. 2, 3 wire	0.320	....	....	0.05213	202.9	1 071
*No. 2	....	0.292	0.292	0.05213	204.9	1 082
No. 3, 3 wire	0.285	....	....	0.04134	160.9	849.6
*No. 3	....	0.260	0.260	0.04134	162.5	858.1
No. 4, 3 wire	0.254	....	....	0.03278	127.6	673.8
*No. 4	....	0.232	0.232	0.03278	128.9	680.5
*No. 5	....	....	0.206	0.02600	102.2	539.6
*No. 6	....	....	0.184	0.02062	81.05	428.0
*No. 7	....	....	0.164	0.01635	64.28	339.4
*No. 8	....	....	0.146	0.01297	50.97	269.1
*No. 9	....	....	0.130	0.01028	40.42	213.4
*No. 10	....	....	0.116	0.008155	32.06	169.3
*No. 12	....	....	0.0915	0.005129	20.16	106.5
*No. 14	....	....	0.0726	0.003225	12.68	66.95
*No. 16	....	....	0.0576	0.002028	7.974	42.10
*No. 18	....	....	0.0456	0.001276	5.015	26.48
*No. 20	....	....	0.0363	0.0008023	3.154	16.65

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* The sizes of cable which have been marked with a single asterisk provide for one or more schedules of preferred series, and are commonly used in the industry. The sizes not marked are given simply as a matter of reference, and it is suggested that their use be discouraged.

[a] To calculate the nominal diameters of class C or class D cables or of any concentric-lay-stranded cables made from round wires of uniform diameters, multiply the diameter of an individual wire (as given in Table II) by that one of the following factors which applies:

Number of Wires in the Cable	Factor to Calculate Cable Diameter
3	2.155
7	3
12	4.155
19	5
37	7
61	9
91	11
127	13
169	15
217	17
271	19

Section 1 (a).—Change to read as follows by the addition of the italicized words and the omission of the word in brackets:

(*a*) These specifications cover [bare,] *concentric-lay*-stranded cables made from round copper wires, for general use as electrical conductors.

Section 4 (c).—Add the following new sentence at the end of this paragraph:

The lay of the layers other than the two outer layers shall be at the option of the manufacturer, unless otherwise agreed upon.

Table II.—In the column headed "Area of Cross-Section, cir. mils" omit the asterisk reference from 1,300,000; insert an asterisk before 1,250,000.

In the column headed "Diameter of Wires" under class D, change the wire size of the 1,700,000 cir. mils cable from "88.6 mils" to read "88.5 mils."

In the column headed "Diameter of Wires" under class B, change the wire size for the 3,500,000 cir. mils cable from "143.8 mils" to read "143.9 mils."

Section 6 (d).—Change from its present form to read as follows:

(*d*) At the option of the purchaser, tension and elongation tests on hard-drawn and medium-hard-drawn wires before stranding may be waived, and the completed hard-drawn and medium-hard-drawn cables may be tested as a unit. The breaking strength of the bare cables so tested shall be at least 90 per cent of the total of the specified minimum breaking strengths of the component wires. The maximum breaking strength of cable made from medium-hard-drawn wires shall be not greater than the sum of the specified maximum breaking strengths of the component wires. The minimum breaking strength of wires shall be calculated using nominal specified diameters and minimum specified tensile strengths. The maximum breaking strength of wires shall be calculated using nominal diameters and maximum specified tensile strengths. The free length between grips of the test specimen shall be not less than 24 in., and care shall be taken to insure that the wires in the cable are evenly gripped during the test (Note 4).

Section 8.—Change from its present form to read as follows:

8. The weight and electrical resistance of a stranded conductor are greater than the total of these characteristics of the wires composing the cable, depending upon the lay or pitch. The standard increment of increase of weight and resistances shall be taken as follows:

Type of Cable	Increment of Increase of Resistance and Weight, per cent
Class AA, Sizes 1 to 4 A.w.g., incl.	1
Classes AA,[a] A, B, C, and D, 2 000 000 cir. mils and under	2
Over 2 000 000 to 3 000 000 cir. mils	3
Over 3 000 000 to 4 000 000 cir. mils	4
Over 4 000 000 to 5 000 000 cir. mils	5

[a] Not less than No. 0 A.w.g.

In cases where the lay is definitely known the increment may be calculated if desired.

Section 9.—At the end of this section add a reference to Note 9.

Explanatory Notes.—Add a new Note 9 to read as follows, including a new Table IV (the accompanying Table I):

NOTE 9.—For the convenience of the users of these specifications Table IV (the accompanying Table I of this report) has been prepared giving the approximate diameters, areas, and weights per 1000 ft. and per mile of the various constructions referred to in Table II.

This recommendation has been submitted to letter ballot of the committee which consists of 41 members; 38 members returned their ballots, of whom 32 have voted affirmatively, 0 negatively, and 6 members marked their ballots "not voting."

The committee recommends lowering the resistivity requirements of the Standard Specifications for Soft or Annealed Copper Wire (B 3 – 39),[3] and for Hot-Rolled Copper Rods for Electrical Purposes (B 49 – 39)[3] to correspond to compensating resistivity changes being considered concurrently by Committee B-2 in the Standard Specifications for Lake Copper Wire Bars (B 4 – 27), and for Electrolytic Copper Wire Bars (B 5 – 27). Approval is being asked on the basis of Committee B-2 approving this change.[4]

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[3] 1939 Book of A.S.T.M. Standards, Part I, pp. 559 and 548.

Resistivity.—In Section 4 of Specifications B 3 and Section 5 (*a*) of Specifications B 49 change the resistivity value from "891.58" to read "875.20" ohms (mile, pounds).

This recommendation has been submitted to letter ballot of the committee which consists of 41 members; 38 members returned their ballots, of whom 31 have voted affirmatively, 1 negatively, and 6 members marked their ballots "not voting."

NEW TENTATIVE STANDARD

Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors.[5]—A draft of these specifications appeared in the preprint of the 1940 report of the committee, but was withdrawn and resubmitted to Subcommittee VIII in view of certain objections raised at the meeting in Atlantic City. A revised draft of the specifications, as appended hereto,[1] has now been presented to the committee and is recommended for publication as tentative.

This recommendation has been submitted to letter ballot of the committee which consists of 41 members; 38 members returned their ballots, of whom 30 have voted affirmatively, 0 negatively, and 8 members marked their ballots "not voting."

This report has been submitted to letter ballot of the committee which consists of 41 members; 38 members have returned their ballots, of whom 37 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

G. E. DEAN,
Secretary.

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[4] See Editorial Note, p. 168.

[5] See Editorial Note below.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-1 presented to the Society through Committee E-10 on Standards, an Emergency Alternate Provision in the Tentative Specifications for Rope-Lay-Stranded and Bunch-Stranded Copper Cables for Electrical Conductors (B 158 – 41 T). This recommendation was accepted by Committee E-10 on November 28, 1941, and the Emergency Alternate Provision appears in the form of an insert sheet in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 369.

REPORT OF COMMITTEE B-2

ON

NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys held one meeting during the year: on June 26, 1940, in Atlantic City, N. J.

Subsequent to the annual meeting in 1940, Committee B-2 presented to the Society through Committee E-10 on Standards new Tentative Specifications for Soft Solder Metal (B 32 – 40 T) as a revision of and to replace immediately the Standard Specifications for Solder Metal (B 32 – 21). This recommendation was accepted[1] by Committee E-10 on August 28, 1940, and the new tentative specifications appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 442.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (J. L. Christie, chairman).—At the meeting of this subcommittee held in Atlantic City, N. J., on June 25, 1940, the Tentative Specifications for Electrolytic Cathode Copper (B 115 – 38 T) were discussed in detail; the comments and criticism being about the same as those made when these specifications were being drafted. It was voted at the meeting to allow these specifications to remain in the tentative status for another year. The question of adopting the specifications as standard was again brought to the attention of the members of the subcommittee by letter ballot on April 7, 1941. Of the 34 members, 22 members returned their ballots, of whom; 19 voted affirmatively, 1 negatively, and 1 "not voting," whereas one member voted in favor of modifying the specifications. The subcommittee has accordingly recommended to Committee B-2 that these specifications be approved and submitted to the Society for formal adoption as standard.

A subgroup has been appointed to prepare specifications covering oxygen-free copper for electrical purposes.[2] Work on this project is now under way under the chairmanship of Carter S. Cole.

Subcommittee III on White Metals and Alloys (G. H. Clamer, chairman).—As mentioned earlier in this report the revised Specifications for Soft Solder Metal (B 32 – 40 T) were accepted as tentative by Committee E-10 on Standards. Some minor revisions have been suggested and are under consideration. The Standard Specifications for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 26) have been under consideration by a subgroup and definite recommendations as to revision are being studied.

Subcommittee VII on Refined Nickel and High Nickel Alloys, Cast and Wrought (O. B. J. Fraser, chairman *pro tem*).—Mr. C. E. Margerum, who has been chairman of Subcommittee VII since its inception, has found it necessary to resign due to the pressure of his official duties at the Bureau of

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-2 reported the following results of the letter ballot vote of a total of 76 ballots returned from a committee membership of 101: Affirmative 44, negative 2, and 30 ballots marked "not voting."

[2] See Editorial Note, p. 168.

Ordnance, United States Navy. The chairman of Committee B-2, therefore, asked O. B. J. Fraser to act as chairman pro tem, pending the election of a permanent subcommittee chairman.

In view of the work under way on numerous specifications, several users of high nickel alloys, already members of the Society, were asked to associate themselves with this effort, and the membership of the committee was correspondingly expanded, with the approval by letter ballot of the B-2 Advisory Committee.

Two meetings of the subcommittee were called, one for New York on February 17 and one for March 5 in Washington. At the former meeting a quorum was not present, but considerable discussion laid the ground work for later action.

At the second meeting, the subcommittee accepted the following changes in the Tentative Specifications for Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 – 39 T):[3]

Table I.—Change the tensile strength ranges of annealed cold-rolled sheet and annealed cold-rolled strip from "70,000 to 80,000" to read "70,000 to 85,000" psi.

Change the tensile strength ranges of quarter-hard cold-rolled sheet and quarter-hard cold-rolled strip from "75,000 to 85,000" to read "78,000 to 85,000" psi.

Change the minimum Rockwell hardness of hard, stress equalized cold-rolled strip from "94B" to "98B."

Section 10 (c).—Change from its present form:

> (*c*) Tension test specimens shall be taken from finished material and in the direction of rolling.

to read as follows:

> (*c*) Tension test specimens from plate, sheet, and strip shall be taken and tested transversely to the direction of rolling, when width will permit.

Table II.—Change from its present form to read as shown in accompanying Table I in order that the tolerances will conform to those given in the Federal Specification for Nickel-Copper Alloy; Forgings, Plates, Rods, Shapes, Sheets, Strips, and Wire (QQ-N-281) (May 28, 1940).

Table III.—Change from its present form to read as shown in accompanying Table II.

TABLE I.—PERMISSIBLE VARIATIONS IN THICKNESS OF COLD-ROLLED SHEET AND STRIP.

Thickness, in.	Permissible Variations, in.	
	plus	minus
COLD-ROLLED SHEET, HARD OR ANNEALED, UP TO 50 IN. IN WIDTH[a]		
0.034 and under	0.002	0.002
Over 0.034 to 0.062, incl.	0.003	0.003
Over 0.062 to 0.125, incl.	0.004	0.004
Over 0.125 to 0.187, incl.	0.005	0.005
Over 0.187 to 0.218, incl.	0.006	0.006
Over 0.218 to 0.234, incl.	0.007	0.007
Over 0.234 to 0.250, incl.	0.008	0.008
COLD-ROLLED SATIN FINISH SHEET, ALL WIDTHS		
0.030 and under	0.002	0.002
Over 0.030 to 0.043, incl.	0.002	0.004
Over 0.043 to 0.070, incl.	0.003	0.005
Over 0.070 to 0.093, incl.	0.003	0.007
STRIP, UP TO 18 IN. IN WIDTH		
0.012 and under	0	0.0005
Over 0.012 to 0.050, incl.	0.001	0.001
Over 0.050 to 0.093, incl.	0.0015	0.0015
Over 0.093 to 0.125, incl.	0.0025	0.0025
Over 0.125 to 0.156, incl.	0.003	0.003
Over 0.156 to 0.250, incl.	0.004	0.004

[a] Cold-rolled sheet over 50 in. in width shall have a permissible variation in thickness of plus or minus 5 per cent of the nominal weight.

Table IV.—In the last column, change the permissible variations in width for sheet of all thicknesses from "±0.062" to "+0.062" and "−0."

The above revisions have been submitted to Committee B-2 and will be acted on at the June meeting for submission to Committee E-10 on Standards.[2]

The subcommittee has prepared nine new specifications for various wrought forms of high nickel alloys the titles of which are as follows:

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[3] 1939 Book of A.S.T.M. Standards, Part I, p. 1134.

Specifications for:

- Nickel, Nickel-Copper Alloy and Nickel-Chromium-Iron Alloy Seamless Condenser Tubes and Ferrule Stock,
- Nickel-Chromium-Iron Alloy Cold-Drawn Pipe and Tubing,
- Nickel Cold-Drawn Pipe and Tubing,
- Nickel-Chromium-Iron Alloy Rods and Bars,
- Nickel Plate, Sheet and Strip,
- Nickel Rods and Bars,
- Nickel-Chromium-Iron Alloy Plate, Sheet and Strip,
- Nickel-Copper Alloy Rods and Bars, and
- Nickel-Copper Alloy Cold-Drawn Pipe and Tubing.

TABLE II.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Thickness, in.	Permissible Excess in Average Weight of Lots for Widths Given, in., Expressed in Percentage of Nominal Weights							
	48 or under	48 to 60, excl.	60 to 72, excl.	72 to 84, excl.	84 to 96, excl.	96 to 108, excl.	108 to 120, excl.	120 to 136, excl.
¼ to 5/16, excl.	9.5	10.5	12	13.5	15	...	...	...
5/16 to ⅜, excl.	7.5	9	10.5	12	13.5	...	...	...
⅜ to 7/16, excl.	7	7.5	9	10.5	12	13.5	...	...
7/16 to ½, excl.	6	7	7.5	9	10.5	12	...	...
½ to ⅝, excl.	5.5	6	7	7.5	9	10.5	12	...
⅝ to ¾, excl.	4.5	5.5	6	7	7.5	9	10.5	12
¾ to 1, excl.	4	4.5	5.5	6	7	7.5	9	10.5
1 or over	4	4	4.5	5.5	6	7	7.5	9

At the meeting in March, numerous changes were suggested and agreed to in the wording of the above specifications. These changes have been incorporated in the specifications which were approved by letter ballot of the subcommittee and submitted to Committee B-2.

Copies of the specifications have been distributed to the members of Committee B-2 and it is expected that at the meeting to be held during the annual meeting these specifications will be approved for submittal to the Society for publication as tentative through Committee E-10 on Standards.[2]

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman).—This subcommittee has under consideration the adoption as standard of the Tentative Specifications for Phosphor Tin (B 51 – 38 T), for Phosphor Copper (B 52 – 38 T), and for Silicon Copper (B 53 – 38 T).

This report has been submitted to letter ballot of the committee, which consists of 118 members; 54 members returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. E. Thum,
Chairman.

C. E. Swartz,
Secretary.

(See Editorial Note, p. 168.)

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-2 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Nickel Rods and Bars (B 160 – 41 T),
Nickel Cold-Drawn Pipe and Tubing (B 161 – 41 T),
Nickel Plate, Sheet, and Strip (B 162 – 41 T),
Nickel, Nickel-Copper Alloy, and Nickel-Chromium-Iron Alloy Seamless Condenser Tubes and Ferrule Stock (B 163 – 41 T),
Nickel-Copper Alloy Rods and Bars (B 164 – 41 T),
Nickel-Copper Alloy Cold-Drawn Pipe and Tubing (B 165 – 41 T),
Nickel-Chromium-Iron Alloy Rods and Bars (B 166 – 41 T),
Nickel-Chromium-Iron Alloy Cold-Drawn Pipe and Tubing (B 167 – 41 T),
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (B 168 – 41 T),

Revision of Tentative Specifications for:

Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 – 39 T), and
Electrolytic Cathode Copper (B 115 – 38 T).

These recommendations were accepted by Committee E-10 on August 25, 1941, and the A.S.T.M. serial designations indicated above were assigned the specifications. The new and revised tentative standards appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 495, 510, 528, 524, 500, 515, 505, 520, 539, 533, and 367.

Committee B-2 also submitted to Committee E-10 the following three additional recommendations which were accepted on January 17, 1942:

Proposed Tentative Specifications for:

Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes

Tentative Revision of Standard Specifications for:

Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 27), and Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars, (B 5 – 27)

The new tentative specifications have been issued as a separate reprint bearing the designation B 170 – 42 T. The tentative revisions of Standard Specifications B 4 and B 5 will be published in the ASTM BULLETIN No. 115, March, 1942.

REPORT OF COMMITTEE B-3

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held two meetings during the year: one in Atlantic City, N. J., on June 26, 1940, and one in Washington, D. C., on March 5, 1941.

The committee has been active during the past year through its Subcommittees I on Total Immersion, III on Spray Test, VI on Atmospheric Corrosion, and VIII on Galvanic and Electrolytic Corrosion in carrying forward the programs under the sponsorship of these subcommittees.

Committee B-3 has authorized the formation of a subcommittee to be known as Subcommittee V on Statistical Analysis and Test Planning and another to be known as Subcommittee VII on Weather Data. The scope of the latter subcommittee will be to obtain more atmospheric data than is given in the usual weather report.

REVISION OF TENTATIVE STANDARD

The committee recommends that the Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 – 39 T) be revised as recommended by Subcommittee III and continued as tentative.

This recommendation has been submitted to letter ballot of the committee which consists of 60 members; 37 members returned their ballots, of whom 32 voted affirmatively, 1 negatively, and 4 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Total Immersion Test (A. W. Tracy, chairman).—This subcommittee has made total immersion tests on copper in normal solutions of sulfuric acid, sodium chloride, and sodium hydroxide, aerated and non-aerated. These cooperative tests showed excellent agreement in 80 per cent of the results, but further work will be necessary before a standard method of test can be presented.

Subcommittee III on Spray Test (E. H. Dix, Jr., chairman).—Subcommittee III met once during the past year, at which time comments received since the publication of the Tentative Method of Salt Spray Testing of Non-Ferrous Metals (B 117 – 39 T)[1] were considered. As a result of these discussions, the following revisions in Method B 117 were approved:

Section 2 (e).—Change the third and fourth sentences from their present form: namely,

> In normally "dry" climates and in connection with chambers operating at elevated temperatures, the compressed air shall be saturated with water at the operating temperature. The design of the saturator is optional but its construction shall be of glass or other corrosion-resistant material, and it shall deliver to the nozzle air having a relative humidity of at least 90 per cent.

to read as follows:

> The compressed air supplied to the nozzle shall have a relative humidity of at least 90 per cent, which may be attained by passing the air through a saturator of optional design but constructed of glass or other corrosion-resistant material. For chambers operating at an elevated temperature, the air shall be heated to

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 1169.

and humidified at the operating temperature (or slightly above) before being supplied to the nozzle.

Omit Note 1 following Section 2 (*e*) and renumber Note 2 accordingly.

Renumber Note 3 following Section 2 (*g*) as Note 2 and change to read as follows:

NOTE 2.—The most satisfactory way to support specimens during exposure is to fasten them to a wooden strip. This method is particularly advantageous for the exposure of irregularly shaped specimens to be exposed at some predetermined slope. The strip should be at the bottom of the specimen, and the area of the specimen in contact with the strip should be protected against special attack. Suspension from glass rods also is an acceptable method of exposure but is normally used only for tension specimens or flat panels. The specimens may be drilled and strung on the rod or suspended from it by glass hooks, wire of essentially the same composition as the specimen, or string suitably treated (as by waxing) to prevent leaching therefrom of corrosive materials. When panels are exposed at a predetermined slope, the spacing of the racks and the angle of slope shall be such that the entire surface is exposed to the freely-settling mist.

With the inclusion of the above changes, the subcommittee decided to continue Method B 117 as tentative.

The subcommittee decided to canvass the industry as completely as possible during the coming year to learn to what extent salt spray testing was being employed, how many laboratories were following the procedures described in Method B 117, and to gather suggestions for improving the present test procedure or preparing additional methods.

Subcommittee IV on Specifications (E. A. Anderson, chairman).—This subcommittee has been discharged as its functions have been taken over by the newly organized Committee B-8 on Electrodeposited Metallic Coatings.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—The principal activity of Subcommittee VI during the past year has been the study, by a special subgroup of the subcommittee, of the statistical methods used by the committee in analyzing the test data resulting from the removal of test specimens to date. The report of this subgroup appended hereto is included as part of the report of Subcommittee VI for this year.

Because of the expanding needs of the Navy in the past year, the subcommittee found it necessary to move its test rack from the Navy reservation to the Army reservation at Key West, Fla. The new location has substantially the same exposure conditions as the previous location.

Repairs are being made to the test racks and supporting structures at several locations where severe corrosion and damage has occurred. The committee has not as yet decided on the date for the next removal of test specimens from the exposure test locations.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman).—This subcommittee has been engaged in assembling specimens and preparing plans for the atmospheric exposure tests on stainless steel coupled with other metals.

The following stainless steels have been selected for this test:

- Stainless steel 18 per cent chromium, 8 per cent nickel—Type 304, and
- Stainless steel 18 per cent chromium, 8 per cent nickel, with 2 to 3 per cent molybdenum—Type 316.

The other metals to be coupled with stainless steel are as follows:

- Aluminum 2S, half hard temper,
- Aluminum alloy 24S-T,
- Aluminum alloy 53S-T,
- Copper electrolytic, tough pitch, hard tempered,
- Mild steel,
- Lead, 6 per cent antimony,
- Zinc, prime western,
- Monel metal, and
- Architectural bronze.

The test locations are as follows:

New York, N. Y.
Altoona, Pa.,
State College, Pa.,
Wilmington, N. C., and
La Jolla, Calif.

All of the specimens for the tests have been received, and it is expected to have the test racks assembled and placed on exposure during the early part of the summer.

During the year no further consideration has been given to the corrosion of galvanic couples exposed to sea water. The work to date on these tests consists of preliminary tests made at Wilmington, N. C. Two papers, one entitled "Some Observations of the Potentials of Metals and Alloys in Sea Water," by F. La Que and G. L. Cox,[2] and the other "Controlling Factors in Galvanic Corrosion," by W. A. Wesley,[2] were sponsored by Committee B-3 and included in the 1940 report. Attention is again called to these papers because they constitute a very valuable contribution on the behavior of metal couples in sea water. It is proposed to resume work on the sea water tests as soon as the racks for the present atmospheric tests have been assembled and placed on exposure.

This report has been submitted to letter ballot of the committee which consists of 60 members; 37 members returned their ballots, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

A. W. TRACY,
Secretary.

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 670, 690 (1940).

REPORT OF SUBCOMMITTEE VI ON ATMOSPHERIC CORROSION

REPORT OF SUBGROUP ON STATISTICAL METHODS OF ANALYSIS

In 1931 Subcommittee VI on Atmospheric Corrosion initiated outdoor exposure tests on rolled samples of 24 nonferrous metals and alloys in nine exposure locations. Two 9 by 12-in. test panels were exposed for weight change determinations in each location. In addition, sufficient machined tension test specimens were exposed to permit removal and testing in groups of six for five exposure periods, originally planned to extend over a total interval of 25 yr. A full description of the metals and alloys included in these tests, giving chemical composition, mechanical properties, and microstructure, appears in the 1932 report of Committee B-3.[1]

When these tests were set up the subcommittee contemplated subjecting six specimens of each material from each exposure location to tensile strength and elongation determinations. An additional set of six specimens held in storage for the same length of time was also to be tested. So far in this program, tests have been made after 1, 3, and 6 yr. of exposure and the data have been presented in tabular form in reports of Subcommittee VI.[2]

When the work was initiated no consideration was given to the possibility of using statistical methods of analyzing the test data. At a later date, however, the desirability of using some form of statistical analysis was recognized and a formula[3] was adopted for the estimation of the change in properties due to pure chance. The applicability of this formula to data of the type obtained in the committee's work has been the subject of some discussion and a special subgroup was appointed in 1938 to investigate the whole matter of how best to analyze these data. As a result, this report has been prepared by the subgroup consisting of E. A. Anderson, chairman, A. W. Tracy, and W. E. Campbell.

The work of this subgroup has developed the conclusion that statistical methods are applicable in the interpretation of the present data in spite of the small number of specimens included in each test. It was also concluded, however, that there are certain difficulties in conducting such analysis and this phase of the problem will require further study. The subgroup is not yet ready to make a final report.

To analyze data statistically requires that there be available the averages and the so-called standard deviations. The latter were not made available in the earlier reports of the subcommittee. As part of the work of the subgroup on statistical analysis the tensile strength data for the three completed exposure periods have been retabulated and the standard deviations recalculated. This work was done to eliminate possible errors and to insure uniformity in the

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 214 (1932).

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 234 (1933); Vol. 35, Part I, p. 142 (1935); Vol. 38, Part I, p. 194 (1938).

[3] See Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 144 (1935).

TABLE I.—STATISTICAL ANALYSIS OF TENSILE STRENGTH RESULTS FROM ATMOSPHERIC EXPOSURE TESTS OF NON-FERROUS METALS.[1]

$\bar{X}$ = Average Tensile Strength in pounds per square inch, and
σ = Standard Deviation in pounds per square inch.

		Pittsburgh, Pa.	Altoona, Pa.	State College, Pa.	New York, N. Y.	Sandy Hook, N. J.	Rochester, N. Y.	Key West, Fla.	Phoenix, Ariz.	La Jolla, Calif.	Storage
Material A—Commercial Copper (Tough Pitch) (99.9 + Cu, 0.026 O). **Original: $\bar{X}$ = 35,200, σ = 556.8**											
1 yr.	$\bar{X}$	33 550	33 750	33 880	33 820	33 920	34 100	34 400	34 270	33 980	34 180
	σ	125.8	125.8	219.2	157.2	106.7	152.8	251.7	314.5	134.4	584.3
3 yr.	$\bar{X}$	32 380	32 550	32 920	32 700	33 050	32 950	33 180	33 150	32 450	33 380
	σ	106.8	76.38	167.4	182.6	214.1	197.9	157.3	206.2	345.2	157.3
6 yr.	$\bar{X}$	32 230	31 920	32 800	32 400	32 800	32 720[a]	33 580	33 330	31 680	33 570
	σ	179.6	121.2	152.8	208.2	163.3	160.0	445.1	286.8	279.4	46.90
Material B—Commercial Copper (Phosphorized) (99.9 + Cu, 0.007 P). **Original: $\bar{X}$ = 35,150, σ = 275.4**											
1 yr.	$\bar{X}$	34 500	34 580	34 930	34 650	34 580	34 870	35 220	35 180	34 670	35 380
	σ	321.5	261.3	281.3	221.7	147.0	136.6	194.5	279.8	136.6	168.2
3 yr.	$\bar{X}$	34 650	34 380	35 020	34 170	34 880	35 070	35 220	35 580	34 630	35 680
	σ	304.1	380.7	333.4	169.3	135.2	204.9	194.5	276.6	298.5	313.5
6 yr.	$\bar{X}$	34 410	34 380	35 030	33 960	34 400	34 630	34 970	35 650	33 570	35 460
	σ	121.4	434.7	94.65	219.3	294.4	125.6	334.6	150.0	194.5	184.2
Material C—Copper-Silicon-Manganese Alloy (96 **Cu,** 3 **Si,** 1 **Mn**). **Original: $\bar{X}$ = 59,070, σ = 679.9**											
1 yr.	$\bar{X}$	57 780	57 330	60 070	57 530	57 980	59 570	58 630	59 330	58 530	59 620
	σ	511.6	754.5	1 838	96.29	129.5	1 951	873.3	411.4	1 598	1 836
3 yr.	$\bar{X}$	57 200	56 920	58 680	55 530	56 970	59 330	57 820	58 880	56 630	58 950
	σ	355.9	317.8	334.3	3 083	677.1	1 271	104.9	706.0	407.3	665.2
6 yr.	$\bar{X}$	56 200	53 150	58 430	56 480	56 320	57 180	57 780	58 970	55 820	59 480
	σ	642.9	3 533	309.8	211.6	825.2	204.4	418.5	617.9	896.9	884.2
Material D—Tin Bronze (92 **Cu,** 8 **Sn**). **Original: $\bar{X}$ = 58,930, σ = 3001**											
1 yr.	$\bar{X}$	56 930	57 650	58 770	57 470	57 220	58 070	58 380	58 650	57 270	58 280
	σ	1 056	457.2	393.9	947.9	806.9	987.5	389.6	694.6	596.0	786.4
3 yr.	$\bar{X}$	56 330	56 480	56 750	56 380	56 770	57 650	57 970	57 320	56 930	57 770
	σ	461.1	430.2	871.3	995.9	521.4	1 084	518.2	956.2	585.3	584.7
6 yr.	$\bar{X}$	55 430	55 220	57 030	55 380	56 730	57 450	57 250	58 220	54 820	56 780
	σ	320.3	1 091	793.3	1 210	335.6	1 037	553.0	1 108	760.1	488.3
Material F—Aluminum Bronze (92 **Cu,** 8 **Al**). **Original: $\bar{X}$ = 74,070,[b] σ = 734.9[b]—Pittsburgh to Key West, incl.** **Original: $\bar{X}$ = 82,860, σ = 1133—Phoenix and La Jolla.** Storage set for Key West and La Jolla: 1 yr. $\bar{X}$ = 83 320. σ = 1772. 3 yr. $\bar{X}$ = 83 920. σ = 900.5. 6 yr. $\bar{X}$ = 83 750. σ = 1443.											
1 yr.	$\bar{X}$	73 670	73 180	76 680	72 440	75 040	76 290	75 940	82 970	83 520	75 830[c]
	σ	1 435	647.3	625.4	2 123	1 433	1 125	1 802	1 323	497.5	1 033
3 yr.	$\bar{X}$	70 930	71 470	74 470	71 720	73 120	74 300	75 070	82 380	84 480	77 400[c]
	σ	1 473	474.4	753.9	1 364	2 701	1 575	2 520	1 114	4 994	731.4
6 yr.	$\bar{X}$	73 060	73 920	75 110	72 910	74 530	75 680	76 050	84 700	85 530	77 130[c]
	σ	1 647	1 039	1 364	1 908	1 081	1 423	1 675	690.4	5 002	969.8
Material G—Commercial Aluminum (99 + **Al**). **Original: $\bar{X}$ = 16,410, σ = 68.73**											
1 yr.	$\bar{X}$	16 300	16 460	16 760	16 530	16 840	16 600	16 670	16 770	15 850	16 220
	σ	110.9	160.2	282.1	205.5	250.8	317.2	550.7	397.2	183.4	40.18
3 yr.	$\bar{X}$	16 520	16 280	16 560	16 370	16 500	16 350	16 590	16 700	15 360	16 410
	σ	188.3	197.0	330.8	114.0	28.28	71.45	196.1	452.1	442.7	112.4
6 yr.	$\bar{X}$	16 030	15 870	16 560	15 930	16 220	16 520	16 360	16 630	13 510	16 700
	σ	204.4	124.9	358.0	71.09	215.9	353.1	214.5	177.1	788.6	53.15
Material I—Aluminum-Manganese Alloy (1 **per cent Mn**). **Original: $\bar{X}$ = 20,450, σ = 243.8**											
1 yr.	$\bar{X}$	19 910	19 800	20 400	20 060	20 100	20 350	20 360	20 480	20 200	20 510
	σ	130.9	153.6	187.6	103.8	80.51	194.4	165.2	176.7	189.1	226.0
3 yr.	$\bar{X}$	19 730	19 530	20 240	19 600	20 050	20 370	20 300	20 640	19 070	20 490
	σ	44.72	100.6	120.9	74.52	181.9	102.1	171.4	226.2	429.0	102.5
6 yr.	$\bar{X}$	19 460	19 030	20 280	19 600	20 100	20 290	20 360	20 040	19 200	20 440
	σ	179.0	86.41	78.84	300.4	190.4	158.2	82.73	263.4	345.6	87.32
Material K—Manganese Bronze (High-Tensile-Strength Yellow Brass) (66.4 **Cu,** 19.3 **Zn,** 5.88 **Al,** 4.6 **Fe,** 3.78 **Mn**) **Original: $\bar{X}$ = 122,000, σ = 4,422**											
1 yr.	$\bar{X}$	118 200	111 460	124 620	116 980	133 460	120 690	124 970	124 760	98 550[a]	121 710
	σ	2 826	4 553	5 884	5 648	13 650	6 213	5 965	6 783	11 480[a]	3 241
3 yr.	$\bar{X}$	115 160	109 410	125 310	104 880	123 180	138 850	122 940	120 530	95 340	132 370
	σ	4 717	5 857	5 664	5 118	6 329	4 681	4 176	7 686	9 111	6 695
6 yr.	$\bar{X}$	86 930	86 550	118 700	78 440	96 690	102 530	103 820	117 270	84 200	123 560
	σ	3 617	9 757	5 491	3 903	1 442	2 992	2 047	5 178	5 771	3 114

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[1] Unless otherwise designated, the original data are based on tension tests of 12 specimens and the other data on tension tests of 6 specimens.

TABLE I.—*Continued*

		Pittsburgh, Pa.	Altoona, Pa.	State College, Pa.	New York, N. Y.	Sandy Hook, N. J.	Rochester, N. Y.	Key West, Fla.	Phoenix, Ariz.	La Jolla, Calif.	Storage
Material L—Brass (85 Cu, 15 Zn). Original: $\bar{X}$ = 39,900, σ = 335.5											
1 yr.	$\bar{X}$......	39 460	40 050	40 410	40 560	40 340	40 680	40 530	40 850	40 090	40 750
	σ......	674.8	354.2	532.9	446.0	322.9	467.0	134.6	437.8	208.4	393.8
3 yr.	$\bar{X}$......	39 030	39 690	40 570	39 740	39 900	40 420	40 990	41 710	40 220	40 980
	σ......	719.4	552.3	296.3	308.4	254.9	447.5	405.3	234.1	334.6	472.6
6 yr.	$\bar{X}$......	37 600	38 070	40 380	38 200	39 670	39 730	40 620	41 150	40 050	40 870
	σ......	613.7	381.2	422.3	562.7	292.0	419.3	333.3	442.5	330.4	672.3
Material M—Brass (70 Cu, 30 Zn). Original: $\bar{X}$ = 48,080, σ = 351.2											
1 yr.	$\bar{X}$......	46 880	47 400	48 250	45 850	47 430	48 300	48 630	49 100	46 880	48 520
	σ......	285.8	707.1	629.2	345.2	350.1	173.2	607.6	200.0	861.2	226.0
3 yr.	$\bar{X}$......	44 180	44 680	49 070	43 000	47 020	47 700	48 280	48 630	46 650	48 880
	σ......	362.9	581.7	280.3	486.5	484.2	282.8	313.7	335.5	236.3	662.1
6 yr.	$\bar{X}$......	41 810	40 070	47 510	39 710	44 770	46 660	47 740	48 360	46 380	48 320
	σ......	630.3	263.5	455.3	535.2	569.6	487.5	679.5	671.9	325.4	721.3
Material N—Admiralty Metal (70 Cu, 29 Zn, 1 Sn). Original: $\bar{X}$ = 54,960, σ = 318.0											
1 yr.	$\bar{X}$......	53 230	53 370	54 230	53 600	53 980	53 920	53 820	53 730	53 580	53 920
	σ......	442.6	308.5	275.5	208.2	291.7	470.1	323.1	335.5	353.7	401.3
3 yr.	$\bar{X}$......	52 320	52 770	53 720	52 520	53 080	53 420	53 470	54 230	53 430	54 200
	σ......	225.9	329.4	463.0	240.2	353.7	463.0	313.9	431.2	138.7	258.2
6 yr.	$\bar{X}$......	50 700	51 520	53 220	51 100	51 770	52 270	53 230	53 770	53 320	54 100
	σ......	369.7	296.2	470.1	104.9	313.9	235.0	112.1	334.5	301.7	305.5
Material O—Duralumin (4 Cu, 0.5 Mn, 0.5 Mg, balance Al). Original: $\bar{X}$ = 63,760, σ = 617.9											
1 yr.	$\bar{X}$......	62 890	58 860	65 910	59 840	59 220	63 560	62 760	64 960	42 950	65 430
	σ......	378.9	3 715	917.0	1 887	2 056	1 134	1 436	631.2	2 490	780.8
3 yr.	$\bar{X}$......	60 870	59 760	63 880	59 790	59 450	62 860	62 930	65 390	43 440	65 170
	σ......	734.4	700.3	1 136	919.9	4 124	351.7	949.3	626.2	2 059	420.6
6 yr.	$\bar{X}$......	60 600	60 020	65 080	58 600	60 940	62 820	63 700	65 350	40 740	65 150
	σ......	1 413	963.5	820.9	816.7	320.6	511.5	935.6	1 148	2 513	285.8
Material P—Copper-Nickel-Zinc Alloy (75 Cu, 20 Ni, 5 Zn). Original: $\bar{X}$ = 54,330, σ = 357.2											
1 yr.	$\bar{X}$......	52 150	53 220	53 480	52 620	53 070	53 720	52 880	53 630	53 500	54 000
	σ......	607.6	421.5	380.9	635.9	303.1	384.3	698.9	263.1	516.4	479.6
3 yr.	$\bar{X}$......	50 970	50 180	52 320	50 820	51 650	52 600	52 580	52 400	52 550	52 680
	σ......	985.8	518.1	392.9	557.7	150.0	454.6	422.4	516.4	734.3	389.5
6 yr.	$\bar{X}$......	50 250	50 470	52 820	49 970	52 020	51 030	52 130	52 950	52 900	53 100
	σ......	125.8	286.2	542.6	1 011	225.9	559.1	293.1	613.1	420.3	447.2
Material Q—Copper-Nickel-Tin Alloy (70 Cu, 29 Ni, 1 Sn). Original: $\bar{X}$ = 76,790, σ = 3803											
1 yr.	$\bar{X}$......	77 270	80 870	78 430	77 550	78 800	78 170	74 870	76 450	77 600	75 750
	σ......	6 514	6 560	5 481	4 630	6 363	2 944	1 990	3 961	3 353	2 917
3 yr.	$\bar{X}$......	78 480	79 630	76 280	74 030	74 550	77 520	74 870	79 320	78 530	77 100
	σ......	5 730	6 107	5 846	4 298	2 477	3 217	3 193	4 785	5 655	2 435
6 yr.	$\bar{X}$......	73 930	74 070	80 020	76 670	75 470	75 650	72 780	78 500	76 280	78 300
	σ......	4 520	4 085	5 649	2 165	4 338	3 916	928.4	5 374	3 503	4 546
Material S—Nickel-Copper Alloy (65 Ni, 32 Cu, 2 Fe, 1 Mn). Original: $\bar{X}$ = 79,390,[d] σ = 1278[d]											
1 yr.	$\bar{X}$......	78 950	78 400	77 670	77 070	79 150	78 500	78 520	79 380	78 670	77 930
	σ......	531.5	1 318	437.8	1 027	1 003	912.9	635.8	853.6	445.4	702.0
3 yr.	$\bar{X}$......	79 180	78 850	79 030	77 000	79 250	79 520	79 970	78 500	79 980	79 150
	σ......	1 053	492.4	415.6	1 069	677.6	933.2	593.0	288.7	628.7	522.0
6 yr.	$\bar{X}$......	78 680	77 720	79 000	76 750	78 030	78 780	79 000	79 050	79 170	79 050
	σ......	1 070	569.4	680.7	797.4	299.0	1 013	611.0	485.6	1 027	377.5
Material T—Commercial Nickel (99 + Ni). Original: $\bar{X}$ = 71,100,[d] σ = 637.4[d]											
1 yr.	$\bar{X}$......	71 000	70 850	71 470	70 560	71 880	71 880	71 280	71 430	71 780	72 050
	σ......	412.3	298.6	799.0	792.1	280.3	618.0	667.2	315.2	837.8	967.3
3 yr.	$\bar{X}$......	69 500	69 230	72 550	70 450	72 270	72 130	72 450	72 970	72 480	73 080
	σ......	1 884	699.5	713.6	987.8	324.2	496.0	752.2	422.4	518.2	227.4
6 yr.	$\bar{X}$......	68 670	67 320	73 650	67 630	72 030	71 530	72 930	72 800	72 900	73 000
	σ......	430.2	480.6	403.1	605.0	364.3	553.2	359.7	300.0	439.7	611.0

TABLE I.—*Concluded*

		Pittsburgh, Pa.	Altoona, Pa.	State College, Pa.	New York, N. Y.	Sandy Hook, N. J.	Rochester, N. Y.	Key West, Fla.	Phoenix, Ariz.	La Jolla, Calif.	Storage
Material U—Chemical Lead (99.92 **Pb**, 0.06 **Cu**). **Original:** $\bar{X}$ = 2581, σ = 54.80											
1 yr.	$\bar{X}$	2 392	2 421	2 429	2 391	2 387	2 488	2 453	2 502	2 361	2 338
	σ	20.73	18.87	29.32	20.62	36.72	53.34	27.77	29.56	14.80	28.45
3 yr.	$\bar{X}$	2 335	2 323	2 345	2 332	2 342	2 397	2 357	2 350	2 357	2 310
	σ	18.93	17.45	7.64	22.32	11.47	13.15	8.56	20.82	16.53	16.33
6 yr.	$\bar{X}$	2 367	2 338	2 375	2 382	2 373	2 366	2 357	2 398	2 392	2 298
	σ	2.54	11.38	23.63	20.77	23.90	17.44	20.97	20.73	15.21	25.74
Material Z—Aluminum-Magnesium-Silicon Alloy (98 **Al**, 0.5 **Mg**, 1 **Si**). **Original:** $\bar{X}$ = 39,980, σ = 835.8											
1 yr.	$\bar{X}$	39 410	38 180	40 210	38 230	39 140	40 100	40 020	41 200	32 210	40 450
	σ	391.2	2 163	203.4	873.1	400.4	433.7	357.5	265.4	1 361	143.5
3 yr.	$\bar{X}$	38 820	38 050	40 050	37 920	39 280	40 780	40 640	43 000	31 370	41 160
	σ	374.2	396.9	328.7	786.7	530.9	855.7	163.9	1 114	1 668	342.0
6 yr.	$\bar{X}$	38 450	36 900	41 310	37 520	37 990	39 810	40 960	43 300	30 200	42 170
	σ	690.5	1 040	576.3	1 181	951.8	496.0	777.2	281.3	1 898	83.26
Material AA—Commercial Zinc (Prime Western Grade, 99 Zn, 0.85 Pb). Original: $\bar{X}$ = 29,940, σ = 298.2											
1 yr.	$\bar{X}$	27 920	28 670	29 000	28 500	29 100	29 580	29 670	29 970	26 560	29 600
	σ	362.2	344.5	216.0	244.9	223.6	279.7	213.0	148.4	674.1^{a}	200.0
3 yr.	$\bar{X}$	27 580	27 200	28 600	27 350	28 270	28 830	29 270	29 300	27 200	29 530
	σ	484.7	182.6	270.8	354.7	220.7	153.6	508.6	305.5	244.9	309.4
6 yr.	$\bar{X}$	25 690	24 880	27 770	25 450	27 570	27 680^{e}	28 470	28 570	23 600^{f}	29 380
	σ	157.8	122.0	73.28	138.4	93.30	82.92^{e}	159.3	109.7	....	90.84
Material BB—Commercial Zinc (High Grade, 99.9 + Zn). Original: $\bar{X}$ = 18,280, σ = 148.0											
1 yr.	$\bar{X}$	18 100	18 150	18 360	18 200	18 400	18 470	18 720	18 680	17 760^{a}	18 580
	σ	115.5	78.38	142.0	0.0	81.65	73.71	89.05	90.44	80.00^{a}	69.61
3 yr.	$\bar{X}$	17 570	17 580	17 900	17 400	18 070	18 050	18 280	18 400	17 150	18 330
	σ	93.66	90.40	264.6	115.5	45.84	95.74	134.8	100.0	125.8	125.2
6 yr.	$\bar{X}$	16 550	15 900	17 950	16 480	17 420	17 400^{a}	17 770	18 120	16 300	18 800
	σ	50.00	152.7	125.8	69.51	67.87	0.0^{a}	110.0	67.83	387.3	152.7
Material CC—Lead-Antimony Alloy (1 per cent Sb). Original: $\bar{X}$ = 3482, σ = 75.84											
1 yr.	$\bar{X}$	3 351	3 273	3 309	3 235	3 230	3 265	3 304	3 274	3 219	3 214
	σ	82.81	35.84	120.0	55.48	23.64	65.46	78.54	36.98	28.64	33.42
3 yr.	$\bar{X}$	3 223	3 152	3 160	3 187	3 125	3 165	3 110	3 073	3 157	3 232
	σ	40.95	25.02	35.12	21.63	17.08	26.30	32.15	16.61	17.35	18.05
6 yr.	$\bar{X}$	3 102	3 062	3 137	3 133	3 117	3 082	3 087	3 022	3 078	3 148
	σ	15.05	23.67	31.64	26.64	28.89	29.33	26.49	32.05	28.30	41.25
Material EE—Commercial Tin (99.85 + Sn). Original: $\bar{X}$ = 3759,^{g} σ = 143.4^{g}											
1 yr.	$\bar{X}$	3 335	3 528	3 530	3 423	3 435	3 453	3 530	3 360	3 270	3 393
	σ	137.0	114.9	118.0	109.0	121.2	62.01	108.3	100.0	28.28	117.5
3 yr.	$\bar{X}$	3 222	3 040	3 158	3 112	3 045	3 168	3 082	3 085	2 888	3 258
	σ	85.79	27.69	33.19	48.23	38.62	73.04	103.6	79.74	216.8	92.75
6 yr.	$\bar{X}$	3 035	2 940	3 157	3 062	2 945	3 145	3 002	2 997	2 788	3 450
	σ	112.2	78.31	65.33	72.76	83.42	44.25	46.48	25.21	73.93	45.09
Material FF—Aluminum-Coated Duralumin. Original: $\bar{X}$ = 58,790, σ = 657.7											
1 yr.	$\bar{X}$	58 910	59 140	58 640	59 280	59 340	59 030	60 240	59 570	59 540	59 530
	σ	757.1	554.1	1 189	826.4	685.7	949.0	560.0	885.9	1 179	484.6
3 yr.	$\bar{X}$	58 490	57 910	59 140	58 740	57 380	58 620	58 390	58 700	58 720	59 300
	σ	633.8	641.5	820.0	850.1	1 083	330.0	935.4	984.2	617.4	921.4
6 yr.	$\bar{X}$	58 840	58 960	59 500	59 260	58 950	57 020	59 860	59 080	59 560	59 470
	σ	693.3	503.5	868.2	714.0	956.1	3 222	800.2	1 009	510.8	819.8
Material HH—Commercial Zinc (Special High Grade 99.99 + per cent Zn). Original: $\bar{X}$ = 18,280,^{h} σ = 167.7^{h}											
1 yr.	$\bar{X}$	18 100	17 470	18 230	17 980	18 330	18 460	18 600	18 700	17 850	18 570
	σ	261.4	203.1	225.0	186.7	90.43	148.8	147.2	57.73	214.1	159.5
3 yr.	$\bar{X}$	17 330	17 360	18 360	17 510	18 290	18 380	18 400	18 490	17 080	18 430
	σ	249.7	134.0	102.3	186.1	123.4	80.36	219.8	171.4	146.6	134.8
6 yr.	$\bar{X}$	16 300	16 100	18 280	16 530	18 160	18 100	18 640	18 690	16 370	19 120
	σ	330.4	210.2	265.8	219.5	127.7	248.3	133.1	229.6	1 091	205.2

[a] 5 specimens.
[b] 10 specimens.
[c] Storage set for first 7 locations, 4 specimens.
[d] 11 specimens.
[e] 4 specimens.
[f] 1 specimen.
[g] 11 specimens.
[h] 6 specimens.

methods of calculation, particularly from the standpoint of rounding off significant figures. These recalculated tensile strength data are presented as a part of this report. The subcommittee is not yet ready to attempt a detailed analysis of the data but feels that publication, particularly of the statistical constants, at this time will make the available results of greater value to engineers interested in corrosion.

No attempt was made to recalculate the elongation data. Careful examination of the available figures indicates that the elongation figures are too erratic for use other than as a broad check on conclusions reached from the tensile strength data.

No decision has been reached by the subcommittee as to when the next set of test specimens will be removed from the exposure racks for test. It is expected, however, that the whole matter of statistical analysis will have progressed sufficiently far by that time to permit a detailed analysis.

The arithmetic means of the tensile strengths in pounds per square inch and the standard deviations for the initial tests and the 1-, 3-, and 6-yr. atmospheric exposure tests are given in Table I. The arithmetic means are given to four significant figures but for calculating the standard deviations arithmetic means to two decimal places were used. The standard deviations, σ, were calculated using the following formula:[4]

$$\sigma = \sqrt{\frac{\text{Sum of } X^2}{n} - \left(\frac{\text{Sum of } X}{n}\right)^2}$$

where:

X = tensile strength of individual specimen, and

n = number of specimens in a group.

Unless otherwise designated, the original data are based on tension tests of twelve specimens and the other data on tension tests of six specimens.

The top line of figures in any one group are the arithmetic means, $\bar{X}$, and the figures underneath are the standard deviations, σ.

Respectfully submitted on behalf of the subcommittee,

W. H. FINKELDEY,
Chairman.

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[4] A.S.T.M. Manual on Presentation of Data, p. 14, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 466 (1933); also issued as separate publication, Third Printing August, 1940, Reprinted March 1941.

REPORT OF COMMITTEE B-4

ON

ELECTRICAL-HEATING, ELECTRICAL-RESISTANCE, AND ELECTRIC-FURNACE ALLOYS

Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys held two meetings during the year: in Columbus, Ohio, on October 30 and 31, 1940, and in New York City on February 20 and 21, 1941, in addition to the meeting held during the annual meeting of the Society.

The committee is cooperating with the Joint A.S.M.E.-A.S.T.M. Research Committee on Effect of Temperature on the Properties of Metals, and with the Alloy Casting Institute in the study of the behavior of metals at high temperatures and in the preparation of methods of testing.

Arrangements have been made for publication by the Society, in about September of this year, of a compilation of the A.S.T.M. standards under the jurisdiction of Committee B-4. There will appear in this publication a paper by W. F. Roeser and H. T. Wensel on "Methods of Testing Thermocouples and Thermocouple Materials" which has been published as a research paper by the National Bureau of Standards.[1]

The committee consists of 53 members, of whom 25 are classified as producers, 17 as consumers, and 6 as general interests.

I. New Tentative Standards

The committee recommends that the following three new methods be accepted for publication as tentative:[2]

Methods of:

Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring-Back Method),

Testing Lateral Wire for Grids of Electronic Devices, and

Testing Wire for Supports Used in Electronic Devices and Lamps.

II. Revisions of Standards

The committee recommends for immediate adoption, revisions in the following specifications as indicated below, and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Elecirical Heating Elements (B 82 - 39):[3]

Sections 7, 8, and 9.—Change the term "specific resistance" to read "resistivity" wherever it appears in these sections.

Section 10.—Change the table from its present form to read as follows:

Temperature	Resistance, per cent
77 F. (25 C.)	100.0
200 F. (93 C.)	100.6
400 F. (204 C.)	101.4
600 F. (315 C.)	102.1
800 F. (427 C.)	102.8
900 F. (473 C.)	103.0
1000 F. (539 C.)	102.7
1200 F. (649 C.)	101.6
1400 F. (760 C.)	101.3
1600 F. (871 C.)	101.5
1800 F. (982 C.)	102.1
2000 F. (1093 C.)	102.8

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[1] *Journal of Research,* Nat. Bureau Standards, Vol. 14, March, 1935, p. 247 (*Research Paper RP768*).

[2] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 547, 549, and 552.

[3] 1939 Book of A.S.T.M. Standards, Part I, p. 687.

Section 12.—Change the last sentence in Paragraph (*a*) from its present form to read as follows by the addition of the italicized words and the omission of those in brackets:

> Test results [shall] *may* be reported *either in hours or in* [as a] percentage of [a reference] *an accepted* standard.

Standard Specifications for Drawn or Rolled Alloy, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron, for Electrical Heating Elements (B 83 - 39):[4]

Section 7, 8, and 9.—Change the term "specific resistance "to read "resistivity" wherever it appears in these sections.

Section 10.—Change the table from its present form to read as follows:

Temperature	Resistance, per cent
77 F. (25 C.)	100.0
200 F. (93 C.)	101.3
400 F. (204 C.)	103.1
600 F. (315 C.)	104.8
800 F. (427 C.)	106.3
1000 F. (539 C.)	107.5
1200 F. (649 C.)	108.0
1400 F. (760 C.)	108.5
1600 F. (871 C.)	109.7

Section 12.—Make the same change in this section as recommended above in Section 12 of Specifications B 82.

III. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the Tentative Method for Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (B 113 - 38 T),[5] revised as indicated below, be approved for reference to letter ballot of the Society for adoption as standard:

Section 1.—Change the second sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets; also omit footnote 2 appearing with sentence:

> It is intended for [use on] *testing round* wire from approximately 0.010 to 0.117 in. (0.25 to 3.00 mm.) in diameter, *and rectangular and flattened wire not over 0.117 in. (3.00 mm.) in thickness where the ratio of width to thickness does not exceed 10.*

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 53 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Temper of Strip and Sheet Metals (Spring-Back Method)	29	0	3
Testing Lateral Wire for Grids of Electronic Devices	29	0	3
Testing Wire for Supports Used in Electronic Devices and Lamps	29	0	3
II. REVISIONS OF STANDARDS			
Spec. for Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Electrical Heating Elements (B 82 - 39), immediate adoption	30	0	2
Spec. for Drawn or Rolled Alloy, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron, for Electrical Heating Elements (B 83 - 39), immediate adoption	30	0	2
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (B 113 - 38 T), as revised	33	0	7

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Life Tests (J. W. Harsch, chairman).—Further tests are being made to determine the effect of ceramics and cements upon the life of heating element materials in contact with them.

Subcommittee II on Electrical Tests (P. H. Dike, chairman).—The work of Subcommittee II has been divided between two sections. Section A on Electrical Tests, (P. H. Dike, chairman) is continuing the work previously carried on by subcommittee II.

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[4] 1939 Book of A.S.T.M. Standards, Part I, p. 690.

[5] 1939 Book of A.S.T.M. Standards, Part I, p. 1156.

Section B on Contact Metals (F. E. Carter, chairman) is developing methods of test for materials for contacts. Five subsections have been organized as follows:

Subsection 1 on Life Tests, B. W. Jones, chairman,

Subsection 2 on Load Carrying Capacity, H. C. Graves, chairman,

Subsection 3 on Standardization of Contact Forms and Sizes, A. B. Gibson, chairman,

Subsection 4 on Research, P. H. Dike, chairman, and

Subsection 5 on Bibliography, S. L. Hoyt, chairman.

Last year life tests were made on contacts with a contacting device used by the Edison General Electric Appliance Co., and also with a device built by the Material Laboratory of the New York Navy Yard in accordance with plans submitted by the committee. After a study of the data from these tests, a preliminary draft of a life test method was prepared. Two contact testing machines were built, one by A. B. Gibson, and the other by the P. R. Mallory Co., in accordance with the proposed method and were carefully studied by the committee in order to combine the best features of each into one machine. The construction of the machine for use in making the life test has been agreed upon. The machine is now being considered for use in making the test for load carrying capacity, so that, if possible, the same equipment may be used for both tests. All of the machines to be used by the cooperating laboratories will be built in one place, and tests will be carried on by the various laboratories during the coming year.

Subcommittee IV on Mechanical Tests (P. H. Brace, chairman).—A proposed Method of Test for Temper of Strip and Sheet Metals (Spring-Back Method)[2] has been prepared at the request of the radio industry and is being submitted as a tentative standard. The temper is determined by measuring the resiliency by the amount of spring-back when flexed. This type of test has been in commercial use with good results for several years.

Subcommittee V on Wrought and Cast Alloys for High Temperature Use (J. W. Harsch, chairman).—The subcommittee has been reorganized, with J. W. Harsch as chairman, and with additional members, both producers and consumers. The significant properties of alloys for use at high temperatures (over 1000 F.) are being studied, and methods of test developed for determining these properties. This work is being carried on in coöperation with the Alloy Casting Institute, and the Joint Committee on the Effect of Temperature on the Properties of Metals.

Preliminary action has been taken as follows:

Properties at Room Temperature:

Tensile Strength.—Tentative Methods of Tension Testing of Metallic Materials (E 8 - 40 T), accepted.

Brinell Hardness.—Standard Method of Test for Brinell Hardness of Metallic Materials (E 10 - 27), accepted.

Impact Strength.—Tentative Methods of Impact Testing of Metallic Materials (E 23 - 34 T), accepted.

Residual (Resultant) Ductility.—This property is an index of structural stability and will be considered with that subject.

Nonmetallic Inclusions.—Awaiting results of study by Committee E-4 on Metallography of methods of determining nonmetallic inclusions.

Basic Test Casting.—The basic test casting developed by the American Casting Institute for research work will be studied to determine its suitability in commercial work for obtaining separately cast specimens.

Properties at Elevated Temperatures:

Short Time Tensile Strength.—Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 - 37 T), accepted tentatively.

Long Time (Creep) Tensile Strength.—Tentative Method of Test for Long-Time (Creep)

High-Temperature Tension Tests of Metallic Materials (E 22 - 38 T), accepted tentatively.

Dilatation (*Thermal Expansivity*).—The Standard Method of Test for Linear Expansion of Metals (B 95 - 39) is being considered.

Rupture (*Time-Temperature Stress, or Accelerated Creep to Rupture*).—A discussion of this subject is being prepared for consideration.

Hardness.—A discussion of this subject is being prepared for consideration.

Structural Stability.—This subject has been referred to a group to review and recommend a program of work.

Fatigue.—Awaiting information requested from the Joint Research Committee on Fatigue of Metals.

Chemical Stability (*Corrosion*).—Referred to Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres to investigate the corrosion of castings in furnace atmospheres.

Thermal Conductivity.—Available methods of determining thermal conductivity will be reviewed.

Warpage.—As warpage depends both upon the properties of the material and the construction of the part, the committee considered that it would be difficult to develop a standard test that would be generally useful.

The committee has kept in mind the fact that methods of test are needed to determine the fundamental properties of the materials as well as control tests for use in purchase specifications. When the methods of test are available, purchase specifications will be prepared.

Subcommittee VI on Specifications (J. W. Harsch, chairman).—The specifications for electrical heater wire and ribbon (B 82 - 39 and B 83 - 39), have been revised to change the requirements for temperature coefficient of resistance to conform to the improved manufacturing practice.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman).—The mechanical problems connected with the determination of elastic properties of thermostat metals at elevated temperatures were considered in considerable detail. It was agreed that the standard method of test should be based on a beam in uniform loading.

A bend test is under consideration to replace the indentation hardness test for determining the temper of thermostat metal. The test is intended as a check on uniformity and not as a performance or fundamental test.

An article describing the properties of thermostat metals has been prepared by S. R. Hood for publication in the compilation of A.S.T.M. Standards on Electrical-Heating, and Resistance, Alloys to be issued this year.[6]

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (A. L. Chapman, chairman).—Two new methods have been completed and are herewith submitted for publication as tentative:[2] namely, Tentative Method of Testing Wire for Supports Used in Electronic Devices and Lamps and Tentative Method of Testing Lateral Wire for Grids in Electronic Devices. A proposed Method of Testing Welds for Incandescent Lamps and Electronic Devices is also being prepared.

Work is being done to develop a method of test for brittleness of tungsten wire. A knife-edge test is being investigated. Purchase specifications are being prepared for round nickel wire for use in electronic devices. Further investigations are being made of various methods for determining the thickness of nickel plating on thin strip steel.

Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman).—Concordant results now have been obtained by four laboratories seeking to reproduce in the laboratory penetrating corrosion of alloys in combusted gas atmospheres. and the formulation of a method of test is being undertaken. Using this test, a study will be made next year of the behavior of various commercial high temperature resistor alloys.

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[6] See "A.S.T.M. Standards on Electrical-Heating and Resistance Alloys," September, 1941, p. 93, available as a separate publication.

This report has been submitted to letter ballot of the committee which consists of 53 members; 32 members returned their ballots, of whom 30 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

DEAN HARVEY,
Chairman.

F. E. BASH,
Secretary.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper Alloys held three meetings during the year: in Atlantic City, N. J., on June 27, 1940, in Washington, D. C., on November 1, 1940, and in Washington, D. C., on March 7, 1941, with an attendance at the three meetings of the committee and its subcommittees of 57, 32 and 34, respectively. In addition to these meetings, the various subcommittees have held meetings quite frequently either in Washington, D. C., New York, N. Y., Philadelphia, Pa., or Waterbury, Conn. during the year to discuss specification requirements of particular interest to the Federal Specifications Board and the War Department.

The following additions and changes in membership have occurred.

J. W. Bolton resigned as first vice-chairman.
H. H. Stout advanced to position of first vice-chairman.
J. J. Kanter elected second vice-chairman.
H. V. Schlacks elected chairman of Subcommittee II to replace R. J. Wheeler who resigned.
R. G. Thompson elected chairman of the newly formed Subcommittee XIII on Tolerances.
A. B. Bagsar, Sun Oil Co., new member.
Sidney Rolle, Scomet Engineering Co., new member.
B. Clements, Wright Aeronautical Corp., new member.
C. H. Hack, National Lead Co., additional representative.
G. E. Behr, National Lead Co., additional representative.
J. S. Rodgers replacing H. Y. Bassett representing Wolverine Tube Co.
W. M. Corse resigned from the Society.
E. L. Lasier, deceased, August 7, 1940.
A. P. Spooner of the Bethlehem Steel Co. to replace P. E. McKinney, deceased.
Raymond Archibald representing the American Association of State Highway Officials to replace R. B. McMinn who has retired; Mr. McMinn had replaced A. L. Gemeny, deceased.
R. S. Pratt, Bridgeport Brass Co., new member and appointed chairman of Subcommittee VII.
J. J. Kanter, named to replace L. A. Ward as chairman of Subcommittee IX.
F. F. Poland, formerly representing the American Smelting and Refining Co., has resigned.
J. E. Schmeltzer, U. S. Maritime Commission, new member.
E. L. Holladay, U. S. Army Ordnance, new member.
R. R. Kennedy, U. S. War Department, Air Corps.
E. C. Forsyth, Standards Branch Bureau of Ships, Navy Department.

The committee lost two of its most active members in the deaths of E. L. Lasier of the U. S. Maritime Commission, and P. E. McKinney of the Bethlehem Steel Co.

At the present time the committee consists of 102 members of which 88 are voting members, 33 are classified as producers, 39 as consumers and 16 as general interest members. A new subcommittee has been organized, designated as Subcommittee XIII on Tolerances. The purpose of this subcommittee is to cooperate with the Copper and Brass Research Association in setting up tolerances for copper and copper-alloy products. The membership of this subcommittee will be limited to consumer representation. It is intended that tolerances, or revisions of existing tolerances, as published by the Copper and Brass Research Association will be suggested to that producer organization. R. G. Thompson of the General Electric Co., Schenectady, N. Y., has been appointed as chairman of this subcommittee.

The membership of the Advisory Committee has been extended to include representatives from the National Bureau of Standards, Navy Department, War Department, and Air Corps. Through this additional membership on the Advisory Committee, it is expected that a much closer cooperation will be obtained between the Federal agencies and the specification writing group of the American Society for Testing Materials.

Changes in the administrative officers of the committee were made necessary due to the resignation of Mr. J. W. Bolton, first vice-chairman, owing to his assumption of the chairmanship of Committee A-3 on Cast Iron. Mr. H. H. Stout, second vice-chairman was advanced to the office of first vice-chairman of the committee, and Mr. J. J. Kanter of the Crane Co. was elected second vice-chairman.

Subsequent to the 1940 annual meeting, Committee B-5 presented to the Society through Committee E-10 on Standards, revisions of the following standard and tentative specifications:

Standard Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 36) (to revert to tentative), and
Tentative Specifications for Seamless Copper Tubes (B 75 – 40 T).

These recommendations were accepted[1] by Committee E-10 on Standards on August 28, 1940. However, in the case of the Tentative Specifications for Seamless Copper Tubes, the recommendations were accepted with the understanding that during the period prior to the 1941 annual meeting further consideration be given to Sections 4 (*b*) and 11.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new specifications and methods be accepted for publication as tentative, as appended hereto:[2]

Tentative Specifications for:

Copper Sheet, Strip, and Plate,
Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes,
Aluminum Bronze Rods, Bars, and Shapes,
Copper-Nickel-Zinc Alloy Rod and Wire,
Manganese Bronze Rods, Bars, and Shapes,
Tin-Bronze and Leaded Tin-Bronze Sand Castings,
High-Leaded Tin-Bronze Sand Castings, to replace the Tentative Specifications of the Alloy: Copper 80 per cent, Tin 10 per cent, Lead 10 per cent (B 74 – 32 T) which are recommended for withdrawal,
Leaded Red Brass and Leaded-Semi Red Brass Sand Castings,
Leaded Yellow Brass Sand Castings for General Purposes,
High-Strength Yellow Brass and High-Strength Leaded Yellow Brass Sand Castings, to replace immediately the Standard Specifications for Manganese-Bronze Sand Castings (B 54 – 39), including the tentative revision published since 1939, which are recommended for withdrawal,
Aluminum Bronze Sand Castings, to replace immediately the Standard Specifications for Aluminum Bronze Castings (B 59 – 39), which are recommended for withdrawal,
Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings, and

Tentative Methods of:

Test for Expansion of Copper and Copper-Alloy Tubing (Pin Test), and

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee B-5 reported the following results of the letter ballot vote from a committee membership of 100: B 30 – 36, affirmative, 37, negative, 0, ballots marked "not voting" 18; B 75 - 40 T, affirmative, 38, negative, 2, ballots marked "not voting" 14.

[2] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 456, 421, 414, 417, 423, 380, 384, 388, 392, 399, 403, 407, 454, 438.

Mercurous Nitrate Test for Copper and Copper Alloys.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends revisions in the following 13 tentative specifications, as given in detail below:

Tentative Specifications for Cartridge Brass, Sheet, Strip, and Disks (B 19 - 40 T):[3]

Dimensional Tolerances.—Revise the tolerances prescribed in Sections 13 to 17, in accordance with the recommendations[4] of Subcommittee XIII on Tolerances (R. G. Thompson, chairman).

Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 40 T):[3]

Table II.—In order to have the chemical requirements for aluminum bronze ingots agree with those in the proposed Tentative Specifications for Aluminum Bronze Sand Castings, change the requirements for grades 9A and 9B from their present form to read as follows:

Classification	Aluminum Bronze			
Numerical Designation	9A		9B	
	Min.	Max.	Min.	Max.
Copper, per cent	78.00	...	88.00	...
Tin, per cent	...	0.50	...	...
Nickel, per cent	...	5.00	...	...
Iron, per cent	2.50	4.50	...	1.25
Aluminum, per cent	7.00	12.00	9.00	11.00
Manganese, per cent	...	3.50	...	...
Other constituents, per cent	...	0.50	...	0.50

Omit from this table footnote *d* which reads as follows: "Maximum impurities including iron but excluding manganese."

Tentative Specifications for Brass Sheet and Strip (B 36 - 40 T):[3]

Table III.—Change the Rockwell hardness values for the superficial 30-T scale for alloy Nos. 5 to 8, inclusive, from their present form to read as follows:

Annealed Temper, Nominal Grain Size	Superficial 30-T Min.	Superficial 30-T Max.
0.120-mm.	..	21
0.070-mm.	12	27
0.050-mm.	20	35
0.035-mm.	25	38
0.025-mm.	27	42
0.015-mm.	33	50

Dimensional Tolerances.—Revise the tolerances prescribed in Sections 13 to 17, inclusive, in accordance with the recommendations[4] of Subcommittee XIII on Tolerances.

Tentative Specifications for Copper Pipe, Standard Sizes (B 42 - 39 T):[5]

Section 4.—Change this section from its present form to read as follows:

4. The copper shall conform to the following requirements as to chemical composition:

Copper,[a] min., per cent	99.90
Phosphorus, max., per cent	0.040

[a] Silver counting as copper.

New Section.—Add a new Section 8 covering Microscopic Examination to read as follows, renumbering the remaining sections accordingly:

8. *Microscopic Examination.*—The pipe shall be made from copper that is free of cuprous oxide as determined by microscopic examination at a 75× magnification.

Tentative Specifications for Seamless Copper Tubing, Bright Annealed (B 68 - 39 T):[5]

Section 2 (a).—Revise the present Paragraph (*a*) as indicated under revision headed "New Section 8," and add a new Paragraph 2 (*a*) to read as follows:

2. (*a*) The tubing shall be made from copper of any one of the three following types:

Type A.—Phosphorized copper, with low residual phosphorus.

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part I.

[4] The revised tables of tolerances have been incorporated in the specifications which appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I.

[5] 1939 Book of A.S.T.M. Standards, Part I.

Type B.—Phosphorized copper, with high residual phosphorus.

Type D.—Oxygen-free copper, without residual metallic deoxidants.

Section 3.—Change the tabulation of chemical requirements from its present form: namely,

Copper, min., per cent	99.90
Phosphorus, max., per cent	0.04

to read as follows:

		Type A	Type B	Type D
Copper,[a] min., per cent		99.90	99.90	99.90
Phosphorus, per cent	min.	0.004	0.015	.....
	max.	0.010	0.040	.....

---

[a] Silver counting as copper.

Section 5 (a).—Change from its present form: namely,

5. (*a*) The tubing shall be thoroughly recrystallized and have a grain size of:
(*1*) 0.015 to 0.040 mm.
(*2*) 0.040 mm. minimum, no maximum.

to read as follows:

5. (*a*) The tubing shall be furnished in either of two annealed tempers as follows:

Temper	Grain Size, mm.
Light anneal	0.015 to 0.040
Soft anneal	0.040 min.

New Section 8.—Change the present Section 2 (*a*) to read as follows, by the addition of the italicized words and the omission of those in brackets, and add as a new Section 8 on Microscopic Examination:

8. *Microscopic Examination.*—[The tubing shall be made from copper that is free from] *Samples of types A, B, and D shall be free of* cuprous oxide as determined by microscopic examination at a 75× magnification.

New Section 9.—Add a new Section 9 covering Embrittlement to read as follows, renumbering the remaining sections accordingly:

9. *Embrittlement.*—It is to be expected that material designated as oxygen-free or deoxidized copper will pass the following embrittlement test, although actual performance of this test is not mandatory under the terms of these specifications, unless definitely specified: Suitable samples shall be heated at a temperature of 800 C. for a period of 20 min. in a furnace containing an atmosphere of hydrogen. After removal from the furnace the samples shall be polished, and examined under a microscope at a 75× magnification. The samples shall not show gassing or open grain structure characteristic of embrittlement.

Tentative Specifications for Seamless Copper Tubes (B 75 – 40 T):[3]

Section 1.—Change from its present form to read as follows:

1. These specifications cover seamless copper tubes suitable for general engineering purposes. Five types are specified, as follows:

Type A.—Phosphorized copper, low residual phosphorus.

Type B.—Phosphorized copper, high residual phosphorus.

Type C.—Phosphorized arsenical copper, high residual phosphorus.

Type D.—Oxygen-free copper without residual metallic deoxidants.

Type F.—Tough pitch arsenical copper.

NOTE.—When tubes are to be welded, the phosphorus deoxidized type should be specified.

Table I.—In order to correct an error in the 1940 printing of these specifications, change the values for grain size for soft annealed and light annealed temper material from "0.40 mm." to read "0.040 mm."

Section 3.—Change from its present form to read as follows:

3. The material shall conform to the following requirements as to chemical composition:

Type	Copper,[a] min., per cent	Phosphorus, per cent		Arsenic, per cent	
		Min.	Max.	Min.	Max.
A	99.90	0.004	0.010	....	....
B	99.90	0.015	0.040	....	....
C	99.40	....	0.040	0.15	0.50
D	99.90	....	....	....	....
F	99.40	....	....	0.15	0.50

[a] Silver counting as copper.

New Section.—Add the following as a new Section 9, renumbering the subsequent sections accordingly:

9. *Electrical Resistivity.*—Types A and D are suitable for electrical conductors. When ordered for this purpose, the electrical resistivity of representative samples of types A and D shall not

exceed the following limits when tested at a temperature of 20 C. (68 F.):

Condition	Electrical Resistivity, max. ohm (meter, gram)
Soft (cold-rolled)	0.15614
Hard (annealed)	0.15940

NOTE.—The International Annealed Copper Standard electrical conductivity equivalents are as follows:

Electrical Resistivity, ohm (meter, gram)	Conductivity, per cent
0.15614	98.16
0.15940	98.16

Section 10.—Omit this section on special tests and add the following two new sections, renumbering the subsequent sections accordingly:

11. *Microscopic Examination.*—The samples of types A, B, C, and D shall be free from cuprous oxide as determined by microscopic examination at 75× magnification.

12. *Embrittlement.*—It is to be expected that types B, C, and D will pass the following embrittlement test, although the actual performance of the test is not mandatory under the terms of these specifications unless definitely specified: Suitable samples shall be heated to a temperature of 800 C. for a period of 20 min. in a furnace containing an atmosphere of hydrogen. After removal from the furnace the samples shall be polished, etched, and examined under a microscope at 75× magnification. The samples shall not show gassing or open grain structure characteristic of embrittlement.

Tentative Specifications for Copper-Silicon Alloy Plate and Sheet (B 96 - 40 T):[3]

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—CHEMICAL REQUIREMENTS.

	Type A	Type C
Copper, min., per cent	94.80	91.00
Silicon, per cent	2.75 to 3.50	2.00 to 3.00
One or more of the following elements may be present within the limits specified below, subject to limits of the specific alloys as given in the Appendix (Table III):		
Manganese, max., per cent	1.50	1.25
Zinc, max., per cent	1.50	4.50
Iron, max., per cent	1.60	0.80
Tin, max., per cent	0.75	...
Lead, max., per cent	0.05	0.05
Nickel, max., per cent	0.60	0.60
Sum of above elements, min., per cent	99.50	99.50

New Section.—Add the following section on minus thickness tolerances:

7. *Minus Thickness Tolerance.*—The thickness of any plate or sheet shall not be more than 0.01 in. under the thickness specified.

Section 8.—Change from its present form to read as follows:

8. *Weight Tolerances.*—The weight of each lot of five or more plates or sheets of the same specified dimensions when ordered to thickness shall not vary from the nominal by more than the amount prescribed in the accompanying Table II. The weight of any individual plate or sheet may vary from the nominal by not more than one third in excess of the tolerance prescribed in the accompanying Table II. The tolerances of lots of less than five plates or sheets shall be governed by the tolerance of individual plates or sheets. For purposes of these specifications, types A and C shall be considered to weigh 0.308 lb. per cu. in.

TABLE II.—LOT OVERWEIGHT TOLERANCES OF PLATE AND SHEET ORDERED TO THICKNESS.

Thickness, in.	Permissible Excess in Average Weight of Lots, Expressed in Percentage of Nominal Weights					
	48 in. and Under in Width	Over 48 to 60 in. in Width	Over 60 to 72 in. in Width	Over 72 to 96 in. in Width	Over 96 to 120 in. in Width	Over 120 to 132 in., incl., in Width
⅛ to 3/16	6.5	8	9	11	..	
Over 3/16 to ¼	6.5	8	9	11	12	
Over ¼ to 5/16	6.5	7.75	8.75	11	12	13
Over 5/16 to ⅜	6.25	7.5	8.5	11	12	13
Over ⅜ to 7/16	6	7.25	8.25	11	12	13
Over 7/16 to ½	6	7	8	10	11	12
Over ½ to ⅝	5.75	6.5	7.5	9	10	11
Over ⅝ to ¾	5.5	6	7	8	9	10
Over ¾ to 1	5	5	6.25	7	8	9
Over 1 to 2	3.5	4	5	6	7	8

Appendix.—Add as an appendix at the end of these specifications, detailed information on commercially available alloy compositions as shown for types A and C alloys in the accompanying Table III.

Tentative Specifications for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 - 40 T):[3,6]

Straightness Tolerances.—Add a new Section 14 on Straightness Tolerances to read as follows, renumbering the remaining sections accordingly:

14. The edgewise curvature (depth of chord) for slit metal in a 72-in. length of metal shall not exceed the following:

Flats	0.5 in.
Coils	1 in.

Tentative Specifications for Leaded Brass Sheet and Strip (B 121 – 39 T):[5]

Section 2 (a).—Change the paragraph headed "Lead" to read as follows by the addition of the italicized words and the omission of those in brackets:

Lead.—[Grade III, common lead of] *Any type conforming to* the Tentative Specifications for Pig Lead (A.S.T.M. Designation: B 29).

Section 3.—In the table change the chemical composition requirements for alloy No. 5 to read as follows by the addition of the italicized figures and the omission of those in brackets:

Copper, per cent	[61.00 to 65.00]	*61.0 to 65.5*
Lead, per cent	[1.75 to 2.25]	*1.5 to 2.5*
Iron, max., per cent	0.10	
Zinc, per cent	remainder	

Table III.—Change the Rockwell hardness values for the superficial 30-T scale for alloy Nos. 2 to 7, inclusive, to read as follows by the addition of the italicized figures and the omission of those in brackets:

Temper, Nominal Grain Size	Superficial 30-T Min.	Superficial 30-T Max.
0.070-mm.	[6] *12*	27
0.050-mm.	[15] *20*	[34] *35*
0.035-mm.	[23] *25*	[41] *38*
0.025-mm.	[28] *27*	[46] *42*

For the information of those using these specifications the commercially available specific alloys are listed in Table III together with the manufacturers' published chemical composition limits. The alloy designations listed below are for convenience only and have no commercial significance.

TABLE III.—COMMERCIALLY AVAILABLE COPPER-SILICON ALLOY SHEET FOR GENERAL PURPOSES.

NOTE.—Analysis shall regularly be made only for the elements specifically mentioned in this table for a designated alloy. If the presence of other elements is suspected, or indicated in the course of routine analysis, further analysis shall be made either chemically or spectrographically to determine that the total of these other elements is not in excess of 0.50 or 0.30 per cent as specifically indicated in the table.

Alloy Designation	Copper	Silicon, per cent	Iron, per cent	Manganese, per cent	Tin, per cent	Zinc, per cent	Lead, max., per cent	Nickel, max., per cent	Copper plus Elements with Specific Limits, min., per cent
				TYPE A					
No. 1	*a*	2.75 to 3.50	0.50 to 1.25	....	....	....	0.05	0.60	99.50
No. 2	*a*	2.75 to 3.25	0.18 max.	....	....	....	0.05	....	99.50
No. 3	*a*	2.75 to 3.25	....	....	0.35 to 0.65	....	0.05	....	99.50
No. 4	*a*	2.75 to 3.25	0.20 max.	....	....	0.50 to 1.50	0.05	....	99.50
No. 5	*a*	2.75 to 3.25	....	0.75 to 1.25	....	....	0.05	....	99.50
No. 6	*a*	2.80 to 3.50	0.80 to 1.60	....	....	....	0.05	....	99.50
No. 7	*a*	2.85 to 3.35	0.25 max.	0.85 to 1.35	....	....	0.05	....	99.50
				TYPE B					
No 11	*a*	0.80 to 1.20	....	....	1.25 to 1.60	....	0.05	....	99.50
No. 12	*a*	1.25 to 1.75	0.25 max.	0.10 to 0.50	....	....	0.05	....	99.50
No. 13	*a*	1.25 to 1.75	0.20 max.	....	....	0.50 to 1.50	0.05	....	99.50
No. 14	*a*	1.50 to 2.00	....	0.15 to 0.40	....	....	0.05	....	99.50
No. 15	*a*	1.75 to 2.25	....	....	0.15 to 0.35	....	0.05	....	99.50
No. 16	*a*	1.50 to 2.00	....	....	....	....	0.05	....	99.50
No. 17	*a*	2.00 to 2.60	0.30 to 0.70	....	....	....	0.05	....	99.70
				TYPE C					
No. 21	*a*	2.00 to 3.00	0.30 to 0.80	....	....	....	0.05	0.60	99.50
No. 22	*a*	2.30 to 3.00	0.30 to 0.70	....	....	....	0.05	....	99.50
No. 23	*a*	2.30 to 3.00	0.30 to 0.70	....	....	2.50 to 4.50	0.05	....	99.50
No. 24	*a*	2.00 to 3.00	0.25 max.	0.65 to 1.25	....	....	0.05	....	99.50

[a] Analysis for copper shall be made in all cases. See last column of this table.

Dimensional Tolerances.—Revise the tolerances prescribed in Sections 13 to

17, inclusive, in accordance with the recommendations[4] of Subcommittee XIII on Tolerances.

Tentative Specifications for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 39 T; ASA H7-1939):[5]

Section 5.—Add the following footnote to the tabulation of dimensional tolerances: "When tolerances are specified as all plus or all minus, double the above values."

Tentative Specifications for Gilding Metal Sheet and Strip (B 130 – 40 T):[3]

Section 1.—Change to read as follows by the addition of the italicized words:

> 1. These specifications cover sheet and strip gilding metal for manufacture of bullet jacket cups *and ammunition components.*

Table II.—Change to read as prescribed in the accompanying Table IV by the addition of the italicized words and figures and the omission of those in brackets:

TABLE IV.—TENSION TEST REQUIREMENTS OF ANNEALED SHEET AND STRIP GILDING METAL. [[a]]

Thickness of Annealed Tempers	Tensile Strength, min., psi.	Elongation in 2 in., min., per cent
Light Anneal:		
[0.020 to 0.050 in.]	[38 000]	[25]
0.005 to 0.010 in.	*38 000*	*15*
0.010 to 0.050 in.	*38 000*	*25*
Over 0.050 to 0.100 in.	38 000	27
Over 0.100 in.	38 000	30
Intermediate Anneal:		
[0.020 to 0.050 in.]	[36 000]	[30]
0.005 to 0.010 in.	*36 000*	*20*
0.010 to 0.050 in.	*36 000*	*30*
Over 0.050 to 0.100 in.	36 000	32
Over 0.100 in.	36 000	35
Drawing Anneal:		
[0.020 to 0.050 in.]	[34 000]	[35]
0.005 to 0.010 in.	*34 000*	*25*
0.010 to 0.050 in.	*34 000*	*35*
Over 0.050 to 0.100 in.	34 000	38
Over 0.100 in.	34 000	40

[[a] On annealed material under 0.020 in. in thickness grain size only shall be specified.]

Dimensional Tolerances.—Revise the tolerances prescribed in Sections 13 to 16, inclusive, in accordance with the recommendations[4] of Subcommittee XIII on Tolerances.

Tentative Specifications for Brass Wire (B 134 – 40 T):[3]

Section 11.—Add the following footnote to the tabulation of tolerances:

> When tolerances are specified as all plus or all minus, double the above values.

Tentative Specifications for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 – 40 T):[3]

Section 5 (a).—Change from its present form to read as follows:

> (*a*) It is recommended that the test specimen coupons be cast to the form and dimensions shown in Fig. 1, and they shall not be chilled. The fin gate along the side shall be not less than $\frac{5}{16}$ in. in thickness at any point along its length.

Section 7 (b).—Change from its present form to read as follows:

> (*b*) The sample for chemical analysis shall be taken by drilling or cutting the test coupon or sprue in such a manner as to be representative of the entire cross-section.

Section 12.—Change from its present form to read as follows:

> 12. *Certification.*—In the case of a product manufactured in advance and supplied for sale from stock by the manufacturer, jobber, or other dealer, the product may upon request of the purchaser be certified by the manufacturer as conforming to these specifications subject to the following procedure: Not less than two tension tests, from different heats, and not less than one chemical analysis shall be made by the manufacturer from each day's melt. Records of the tension test results and chemical analysis shall be systematically made and maintained and shall be the basis for certification. In lieu of the manufacturer's certification and upon written request by the purchaser, these records may be examined by the purchaser or his accredited representative upon the manufacturer's premises.

III. REVISIONS OF STANDARDS

The committee recommends for immediate adoption, revisions in certain specifications, as indicated below, and accordingly asks for the necessary nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for Copper Plates for Locomotive Fireboxes (B 11 – 33):[5]

Section 1.—Change from its present form to read as follows:

1. These specifications cover four grades of copper plates for locomotive fireboxes, as follows:

Grade 1, Tough pitch copper (nonarsenical),
Grade 2, Phosphorized copper (nonarsenical),
Grade 3, Tough pitch arsenical copper, and
Grade 4, Phosphorized arsenical copper.

Section 3.—Change from its present form to read as follows:

3. The copper shall conform to the following requirements as to chemical composition:

	Copper (plus silver), min., per cent	Arsenic, per cent	Phosphorus, per cent
Grade 1	99.90	.....	.....
Grade 2	99.90	.....	0.015 to 0.040
Grade 3	99.40	0.15 to 0.50	.....
Grade 4	99.40	0.15 to 0.50	0.015 to 0.040

Section 9.—Change from its present form to read as follows:

9. The weight of each lot of five or more plates of the same grade and the same specified dimensions when ordered to thickness shall not vary from the nominal by more than the amount prescribed in the accompanying Table V. The weight of any individual plate may vary from the nominal by not more than one-third in excess of the tolerances prescribed in Table V. The tolerance of lots of less than five plates shall be governed by the tolerance of individual plates. One cubic inch of rolled copper is assumed to weigh 0.323 lb.

TABLE V.—LOT WEIGHT TOLERANCES OF PLATES ORDERED TO THICKNESS.

Thickness, in.	Weight Tolerances, plus and minus, Expressed in Percentage of Theoretical Weight				
	48 in. and Under in Width	Over 48 to 60 in. in Width	Over 60 to 72 in. in Width	Over 72 to 90 in. in Width	Over 90 to 110 in., incl., in Width
⅛ and under	8	9.5	11	12.5	14
Over ⅛ to 3/16	6.5	8	9.5	11	12.5
Over 3/16 to ¼	6	7.5	8.5	9	10
Over ¼ to 5/16	5.5	7	8	8.5	9
Over 5/16 to ⅜	5	6	7	7.5	8
Over ⅜ to 7/16	4.5	5	6	7	7.5
Over 7/16 to ½	4	4.5	5.5	6	6.5
Over ½ to ⅝	3.5	4.5	5	5.5	6
Over ⅝ to ¾	3	4	4.5	5	5.5
Over ¾ to 1	2.75	3.5	4	4.5	5
Over 1 to 1½	2.5	3	3.5	4	4.5
Over 1½ to 2, incl.	2.25	2.75	3.25	3.75	4.25

Standard Specifications for Seamless Copper Boiler Tubes (B 13 – 40):[3]

Section 1.—At the end of Section 1 add a note to read as follows:

NOTE.—In the event copper boiler tubes for welding purposes are desired, deoxidized copper should be ordered to the Tentative Specifications for Seamless Copper Tubes (A.S.T.M. Designation: B 75) of the American Society for Testing Materials in so far as chemical composition requirements only are concerned. Other requirements shall be in accordance with these Standard Specifications B 13.

Standard Specifications for Free-Cutting Brass Rod for Use in Screw Machines (B 16 – 29; ASA H8-1934):[5]

The following revisions are recommended in order to include in these specifications requirements for soft and half-hard tempers in accordance with Federal Specification QQ-B-611a:

Section 1.—Reletter as Paragraph (*a*) and add the following as Paragraph (*b*):

(*b*) Half-hard temper shall be supplied unless otherwise specified.

Tensile Properties.—Add a new Section 6 on Tensile Properties to read as follows, renumbering the remaining sections accordingly:

6. The material shall conform to the requirements as to tensile properties prescribed in the accompanying Table VI.

Section 6.—Reletter as Paragraph (*a*) and add the phrase "in the half-hard temper" after "Rods or bars."

Add a new Paragraph (*b*) to read as follows:

(*b*) The bend test may be waived at the option of the purchaser.

Section 8.—Add the following footnote to the tabulation of dimensional toler-

TABLE VI.—TENSILE REQUIREMENTS.

Temper	Nominal Diameter or Distance Between Parallel Faces, in.		Tensile Strength, min., psi.	Yield Strength, min., psi.	Elongation in 2 in., min., per cent
Soft	1 and under		48 000	20 000	20
	Over 1 to 2		44 000	18 000	25
	Over 2		40 000	15 000	30
Half hard (rounds, hexagons, and octagons)	0.50 and under		60 000	28 000	7
	Over 0. 50 to 1		55 000	25 000	15
	Over 1 to 2		50 000	20 000	20
	Over 2		45 000	15 000	25
	Thickness, in.	Width, in.			
Half hard (rectangles and squares)	1 and under	2 and under	50 000	25 000	10
	Over 1	Over 2	45 000[a]	17 000[a]	20[a]

[a] In the case of bars either over 1 in. in thickness or over 2 in. in width, or both, these values shall apply.

ances: "When tolerances are specified as all plus or all minus, double the above values."

Standard Specifications for Copper Water Tube (B 88 – 39; ASA H23.1-1939):[5]

Sections 2 and 4, and Table II.—Omit all references and requirements for type O tubing.

Section 5.—Change from its present form: namely,

> 5. The copper shall have a purity of at least 99.90 per cent as determined by electrolytic assay, silver being counted as copper.

to read as follows:

> 5. The copper shall conform to the following requirements as to chemical composition:

Copper,[a] min., per cent	99.90
Phosphorus, max., per cent	0.04

---

[a] Silver counting as copper.

Table I.—Change the tensile strength requirements and the designations for "light temper" and "drawn temper" to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

Temper	Tensile Strength, psi.
Light *drawn*[a]	[40 000] *36 000* to 50 000
Hard drawn	[40 000] *48 000* min.

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[a] *Light drawn temper applies only to types K and L tubing.*

Standard Specifications for Sheet Copper-Silicon Alloy (B 97 – 40):[3]

Table I.—Change to read as shown in the accompanying Table VII.

TABLE VII.—CHEMICAL REQUIREMENTS.

	Type A	Type B	Type C
Copper, min., per cent	94.80	96.00	91.00
Silicon, per cent	2.75 to 3.50	0.75 to 2.00[a]	2.00 to 3.00
One or more of the following elements may be present within the limits specified below, subject to limits of the specific alloys as given in the Appendix (Table III):			
Manganese, max., per cent	1.50	0.75	1.25
Zinc, max., per cent	1.50	1.50	4.50
Iron, max., per cent	1.60	0.80	0.80
Tin, max., per cent	0.75	1.60	....
Nickel, max., per cent	0.60	....	0.60
Lead, max., per cent	0.05	0.05	0.05
Sum of above elements, min., per cent	99.50	99.50	99.50

[a] An alloy containing as high as 2.60 per cent silicon is acceptable providing the sum of all elements, other than copper, silicon, and iron does not exceed 0.30 per cent.

Straightness Tolerances.—Add a new Section 12 on Straightness Tolerances to read as follows, renumbering the remaining sections accordingly:

> 12. The edgewise curvature (depth of chord) for slit metal in a 72-in. length of metal shall not exceed the following:

Flats	0.5 in.
Coils	1 in.

Appendix.—Add as an appendix at the end of these specifications, detailed information on commercially available alloy compositions as shown in the accompanying Table III.

Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 40)[3] *and Copper-Silicon Alloy Wire for General Purposes (B 99 – 40)*:[3]

Table I.—Change to read as shown in the accompanying Table VIII.

Appendix.—Add as an appendix at the end of these specifications, detailed information on commercially available alloy compositions as shown in the accompanying Table IX.

TABLE VIII.—CHEMICAL REQUIREMENTS.

	Type A	Type B	Type C	Type D
Copper, min., per cent	94.80	96.00	91.00	94.00
Silicon, per cent	2.75 to 3.50	0.75 to 2.00[a]	2.00 to 3.00	2.75 to 3.50
Lead, per cent	....	....	....	0.20 to 0.80
One or more of the following elements may be present within the limits specified below, subject to limits of the specific alloys as given in the Appendix (Table IX):				
Manganese, max., per cent	1.50	0.75	1.25	1.50
Zinc, max., per cent	1.50	1.50	4.50	1.50
Iron, max., per cent	1.60	0.80	0.80	0.25
Tin, max., per cent	0.75	1.60	....	....
Lead, max., per cent	0.05	0.05	0.05	....
Sum of above elements, min., per cent	99.50	99.50	99.50	99.50

[a] An alloy containing as high as 2.60 per cent silicon is acceptable providing the sum of all elements, other than copper, silicon, and iron does not exceed 0.30 per cent.

For the information of those using these specifications the commercially available specific alloys are listed in Table IX together with the manufacturers' published chemical composition limits. The alloy designations listed below are for convenience only and have no commercial significance.

TABLE IX.—COMMERCIALLY AVAILABLE COPPER-SILICON ALLOY WIRE.

NOTE.—Analysis shall regularly be made only for the elements specifically mentioned in this table for a designated alloy. If the presence of other elements is suspected, or indicated in the course of routine analysis, further analysis shall be made either chemically or spectrographically to determine that the total of these other elements is not in excess of 0.50 or 0.30 per cent as specifically indicated in the table.

Alloy Designation	Copper	Silicon, per cent	Iron, per cent	Manganese, per cent	Tin, per cent	Zinc, per cent	Lead, per cent	Copper plus Elements with Specific Limits, min., per cent
TYPE A								
No. 2	[a]	2.75 to 3.25	0.18 max.	....	....	....	0.05 max.	99.50
No. 3	[a]	2.75 to 3.25	....	....	0.35 to 0.65	....	0.05 max.	99.50
No. 4	[a]	2.75 to 3.25	0.20 max.	....	....	0.50 to 1.50	0.05 max.	99.50
No. 5	[a]	2.75 to 3.25	....	0.75 to 1.25	....	....	0.05 max.	99.50
No. 6	[a]	2.80 to 3.50	0.80 to 1.60	....	....	....	0.05 max.	99.50
No. 7	[a]	2.85 to 3.35	0.25 max.	0.85 to 1.35	....	....	0.05 max.	99.50
TYPE B								
No. 11	[a]	0.80 to 1.20	....	....	1.25 to 1.60	....	0.05 max.	99.50
No. 12	[a]	1.25 to 1.75	0.25 max.	0.10 to 0.50	....	....	0.05 max.	99.50
No. 13	[a]	1.25 to 1.75	0.20 max.	....	....	0.50 to 1.50	0.05 max.	99.50
No. 14	[a]	1.50 to 2.00	....	0.15 to 0.40	....	....	0.05 max.	99.50
No. 15	[a]	1.75 to 2.25	....	....	0.15 to 0.35	....	0.05 max.	99.50
No. 16	[a]	1.50 to 2.00	....	....	....	....	0.05 max.	99.50
No. 17	[a]	2.00 to 2.60	0.30 to 0.70	....	....	....	0.05 max.	99.70
TYPE C								
No. 22	[a]	2.30 to 3.00	0.30 to 0.70	....	....	....	0.05 max.	99.50
No. 23	[a]	2.30 to 3.00	0.30 to 0.70	....	....	2.50 to 4.50	0.05 max.	99.50
No. 24	[a]	2.00 to 3.00	0.25 max.	0.65 to 1.25	....	....	0.05 max.	99.50
TYPE D								
No. 31	[a]	2.75 to 3.25	0.20 max.	....	....	0.50 to 1.50	0.25 to 0.50	99.50
No. 32	[a]	2.85 to 3.35	0.25 max.	0.85 to 1.35	....	....	0.30 to 0.75	99.50

[a] Analysis for copper to be made in all cases. See last column of this table.

Standard Specifications for Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60 – 36):[5]

Section 1 (c).—Omit this paragraph which reads as follows:

> (*c*) It is recommended that this alloy shall not be used where castings are subjected to a temperature exceeding 500 F. (260 C.).

Section 4 (*b*).—Make the same change in this section as is recommended above in Section 7 (*b*) of Specifications B 132.

Section 6 (*a*).—Make the same change in this section as is recommended above in Section 5 (*a*) of Specifications B 132.

Figure 2.—Add an additional dimension of $\frac{1}{2}$ in. for the width of web.

New Section.—Add as new Section 12, the certification clause recommended above for inclusion as Section 12 of Specifications B 132.

Standard Specifications for Steam or Valve Bronze Castings (*B 61 – 40*):[3]

Section 1.—Change from its present form: namely,

> 1. These specifications cover alloy castings, the alloy being a composition of copper, tin, lead, and zinc, commonly used as a high-grade steam metal or valve bronze, which is suitable for applications involving temperatures[a] of 500 F. (260 C.).
>
> ---
>
> [a] The temperature recommendation of the scope clause is conservative in view of known design practice. Establishment of definite limiting design stresses and of dimensional standards later may possibly permit some raising of the limiting temperature above that now recommended.

to read as follows:

> 1. These specifications cover alloy castings, the alloy being a composition of copper, tin, lead, and zinc, commonly used as a high-grade steam metal or valve bronze.[a]
>
> ---
>
> [a] The Rules for Construction of Unfired Pressure Vessels of the A.S.M.E. Boiler Construction Code (Par. U-20 (*f*)) require that bronze valves and steam metal conforming to these specifications shall be limited to temperatures of 550 F.

Section 4 (*b*).—Make the same change in this section as is recommended above in Section 7 (*b*) of Specifications B 132.

Section 6 (*a*).—Make the same change in this section as is recommended above in Section 5 (*a*) of Specifications B 132.

Figure 2.—Make the same change in this figure as is recommended above in Fig. 2 of Specifications B 60.

New Section.—Add as new Section 12, the certification clause recommended above for inclusion as Section 12 of Specifications B 132.

Standard Specifications for Composition Brass or Ounce Metal Castings (*B 62 – 36*):[5]

Section 1.—Omit the second sentence which reads as follows:

> Castings of this alloy are commonly used in hydrostatic pressure up to 350 psi.

Add a new footnote to this section to read as follows:

> The Rules for Construction of Unfired Pressure Vessels of the A.S.M.E. Boiler Construction Code (Par. U-20 (*f*)) require that composition or ounce metal conforming to these specifications shall be limited to temperatures of 406 F.

Section 4 (*b*).—Make the same change in this section as is recommended above in Section 7 (*b*) of Specifications B 132.

Section 6 (*a*).—Make the same change in this section as is recommended above in Section 5 (*a*) of Specifications B 132.

Figure 2.—Make the same change in this figure as is recommended above in Fig. 2 of Specifications B 60.

New Section.—Add as new Section 12 the certification clause recommended above for inclusion as Section 12 of Specifications B 132.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the Tentative Specifications for Brass Pipe, Standard Sizes (B 43 – 40 T)[3] be approved without change for reference to letter ballot of the Society for adoption as standard.

V. WITHDRAWAL OF STANDARDS AND TENTATIVE STANDARDS

The committee recommends the withdrawal of the following three standard specifications, one tentative standard, and two existing tentative revisions since these are being superseded by new tentative specifications as mentioned earlier in this report:

Standard Specifications for:

Manganese-Bronze Sand Castings (B 54 – 39), including existing tentative revision,

Aluminum Bronze Castings (B 59 – 39),

Manganese-Bronze Ingots for Sand Castings (B 7 – 39), including existing tentative revision. This standard is being withdrawn since the ingots covered by these specifications are now included in the present Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 40 T), and

Tentative Specifications for Sand Castings of the Alloy: Copper 80 per cent, Tin 10 per cent, Lead 10 per cent (B 74 – 32 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 88 voting members; with the results shown in Table X.

VI. EDITORIAL CHANGE IN STANDARDS AND TENTATIVE STANDARDS

The committee recommends as an editorial matter that a paragraph on Basis of Purchase be added to all standards and tentative specifications under its jurisdiction. In this paragraph will be given the pertinent information necessary for the correct purchase of the material. This editorial change is to be made in each of the specifications at their earliest printing.

ACTIVITIES OF SUBCOMMITTEES

Committee B-5 during the past year has extended the work of its subcommittees by the appointment of individual subgroups or special subcommittees functioning as working committees on a particular problem or assignment. Meetings of these smaller special subgroups have been held during the year in Washington, D. C., Philadelphia, Pa., New York, N. Y., and Waterbury, Conn. The general results of this activity are reported under the particular subcommittees to which they belong.

Subcommittee I on Copper-Zinc Sheet and Strip (G. H. Harnden, chairman) has given special attention to the revision of tables of tolerances, with changes in Rockwell hardness of brass sheet, and to a round-robin study of the methods of grain size determinations. The situation relative to tolerances, however, developed to a point where it was felt that a separate subcommittee responsible for tolerances should be organized. Accordingly Subcommittee XIII on Tolerances has now been organized and further reference to its work appears later in this report.

In the grain size round-robin study several laboratories have been actively cooperating with the Frankford Arsenal, Philadelphia, Pa., and have accumulated considerable data which will ultimately be presented to the Society through Committee B-5. Preliminary analysis has indicated the need for specializing the method of obtaining data on grain size. At the present time new specimens have been prepared by Frankford Arsenal covering various anneals on sheet brass which are to be submitted to a special subcommittee for further study of grain size. The program of investigation has been developed by this special subcommittee in cooperation with representatives of the War Department and statisticians and all data are to be obtained as outlined by this group. Upon completion of the readings, the report is to be summarized and it is anticipated that information will be developed relative to the variation to be expected by an observer on various grain sizes as well as the expectancy that may be obtained between observers on standard grain sizes. Specific forms and instructions have been developed for obtaining these data. The results should permit the setting up of methods of obtaining grain size on copper and copper alloys as well as new standards of grain size limits which should eliminate a great many of the difficulties heretofore experienced in specifications calling for grain size measurements.

TABLE X.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Copper Sheet, Strip, and Plate	34	3	16
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes	35	0	22
Spec. for Aluminum Bronze Rods, Bars, and Shapes	34	1	20
Spec. for Copper-Nickel-Zinc Alloy Rod and Wire	58	0	8
Spec. for Manganese Bronze Rods, Bars, and Shapes	58	0	8
Spec. for Tin-Bronze and Leaded Tin-Bronze Sand Castings,	39	3	12
Spec. for Leaded Red Brass and Leaded Semi Red Brass Sand Castings	39	3	12
Spec. for Leaded Yellow Brass Sand Castings for General Purposes	39	3	12
Spec. for High-Strength Yellow Brass and High-Strength Leaded Yellow Brass Sand Castings, to replace Standard Spec. B 54 - 39 and tentative revision	57	1	8
Spec. for Aluminum Bronze Sand Castings, to replace Standard Specs. B 59 - 39	39	1	14
Spec. for Leaded Nickel Brass (Leaded Nickel Silver) and Leaded Nickel Bronze (Leaded Nickel Silver) Sand Castings	37	2	15
Spec. for High-Leaded Tin-Bronze Sand Castings, to replace Tentative Spec. (B 74 - 32 T)	40	1	13
Test for Expansion of Copper and Copper-Alloy Tubing (Pin Test)	58	0	8
Mercurous Nitrate Test for Copper and Copper Alloys	42	0	13
II. REVISION OF TENTATIVE STANDARDS			
Spec. for Cartridge Brass, Sheet, Strip, and Disks (B 19 - 40 T)	32	0	2
Spec. for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 40 T)	32	0	2
Spec. for Brass Sheet and Strip (B 36 - 40 T)	32	0	2
Spec. for Copper Pipe, Standard Sizes (B 42 - 39 T)	44	1	14
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 39 T)	58	0	8
Spec. for Seamless Copper Tubes (B 75 - 40 T)	43	0	16
Spec. for Copper-Silicon Alloy Plate and Sheet (B 96 - 40 T)	58	0	8
Spec. for Beryllium Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 - 40 T)	32	0	2
Spec. for Leaded Brass Sheet and Strip (B 121 - 39 T)	58	0	8
Spec. for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 39 T; ASA H7-1939)	32	0	2
Spec. for Gilding Metal Sheet and Strip (B 130 - 40 T)	32	0	2
Spec. for Brass Wire (B 134 - 40 T)	32	0	2
Spec. for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 - 40 T)	58	0	8
III. REVISIONS OF STANDARDS			
Spec. for Copper Plates for Locomotive Fireboxes (B 11 - 33), immediate adoption	58	0	8
Spec. for Seamless Copper Boiler Tubes (B 13 - 40), immediate adoption	43	0	16
Spec. for Free-Cutting Brass Rod for Use in Screw Machines (B 16 - 29; ASA H8-1934), immediate adoption	58	0	8
Spec. for Copper Water Tube (B 88 - 39; ASA H23.1-1939), immediate adoption	58	0	8
Spec. for Sheet Copper-Silicon Alloy (B 97 - 40), immediate adoption	58	0	8
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 40), immediate adoption	58	0	8
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 40), immediate adoption	58	0	8
Spec. for Castings of the Alloy: Copper 88 per cent, Tin 8 per cent, Zinc 4 per cent (B 60 - 36), immediate adoption	58	0	8
Spec. for Steam or Valve Bronze Castings (B 61 - 40), immediate adoption	58	0	8
Spec. for Composition Brass or Ounce Metal Castings (B 62 - 36), immediate adoption	58	0	8
IV. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Brass Pipe, Standard Sizes (B 43 - 40 T)	32	0	2
V. WITHDRAWAL OF STANDARDS AND TENTATIVE STANDARDS			
Spec. for Manganese-Bronze Sand Castings (B 54 - 39), including existing tentative revision	22	2	9
Spec. for Aluminum Bronze Castings (B 59 - 39)	24	0	9
Spec. for Manganese-Bronze Ingots for Sand Castings (B 7 - 39), including existing tentative revision	24	0	9
Spec. for Sand Castings of the Alloy: Copper 80 per cent, Tin 10 per cent, Lead 10 per cent (B 74 - 32 T)	32	0	2

Another group of this subcommittee has been working on methods of test that might be satisfactorily employed for obtaining bend test requirements on primer brass. It is anticipated that this work also will ultimately lead to further revisions of the specifications controlling the quality of this material.

The subcommittee is recommending minor changes, as given earlier in this report, in the Tentative Specifications for Brass Sheet and Strip (B 36 – 40 T), Gilding Metal Sheet and Strip (B 130 – 40 T), and Leaded Brass Sheet and Strip (B 121 – 39 T). In these three specifications and also in the Tentative Specifications for Cartridge Brass Sheet, Strip, and Disks (B 19 – 40 T) certain changes in dimensional tolerances are also to be made. This matter is now being studied by Subcommittee XIII on Tolerances in cooperation with the Copper and Brass Research Association.

Subcommittee II on Copper-Tin Sheet and Strip (H. V. Schlacks, chairman) has not been active during the year, inasmuch as the need for revisions in the specifications under its jurisdiction has not been found necessary. During the year, the former chairman, R. J. Wheeler, found it necessary to resign, and Mr. Schlacks was elected in his place. The committee in accepting with regret the resignation of Mr. Wheeler expresses its deep appreciation of his valuable aid during his term of service as chairman.

Subcommittee III on Copper-Nickel-Zinc Sheet and Strip (E. S. Bunn, chairman) has not been active and no changes have been found necessary in the Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 39 T), which are under its jurisdiction.

Subcommittee IV on Miscellaneous Copper Base, Sheet and Strip Alloys (L. A. Ward, chairman) has held a number of meetings and has sponsored quite a few meetings of its subgroups. The Tentative Specifications for Copper-Silicon Alloy Plate and Sheet (B 96 – 40 T) and for Sheet Copper-Silicon Alloy (B 97 – 40) are being revised to allow the inclusion of a requirement for nickel of 0.6 maximum per cent in alloys of types A, B, and C. A new table of compositions of the various copper-silicon alloys covered by Specifications B 96 and B 97 is being appended to the specifications. It is felt that this is desirable inasmuch as there has been a strong feeling among the members of the committee to the effect that the grouping of a large number of alloys under one broad range of chemical composition is undesirable. This is a reversal of the trend of committee actions for about the past six or eight years. A special subgroup has been obtaining data on the tolerances for hot-rolled material covered by Specifications B 96, and, on the basis of these data, it was found desirable to maintain the present tolerances in force for at least one additional year.

The committee has actively cooperated with the Federal Specifications Board and has recommended certain changes in the Federal Specification for Copper-Silicon-Alloy Bars, Plates, Rods, Shapes, Sheets, and Strips (QQ-C-591). Correlation of the requirements in the Federal Specifications and the corresponding A.S.T.M. Specifications has been successfully carried out.

The subcommittee has also given special study to the Tentative Specifications for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 – 40 T), and in cooperation with the Aeronautical Specifications Board, is planning a change in the requirement for the beryllium content from "2.00 to 2.25 per cent" to "1.90 to 2.20 per cent." This change will probably be presented to the Society through Committee E-10 on Standards later in the year.[6] The addition of straightness tolerances to Specifications B 120 has been recommended by the subcommittee.

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[6] See Editorial Note, p. 198.

Another subgroup reported on sizes of phosphorized cakes available from refineries. A final report and recommendations are to be submitted to Subcommittee IV in June.

Proposed Specifications for Copper Sheet, Plate, and Strip have been prepared as mentioned earlier in this report.

Subcommittee V on Miscellaneous Copper-Base Wire and Rod Alloys (W. H. Bassett, Jr., chairman) has completed the preparation of specifications for manganese-bronze rod, aluminum-bronze rod, and leaded red-brass rod, as mentioned earlier in this report, which are being recommended for publication as tentative. Two other specifications which have been completed, covering phosphor-bronze rod and phosphor-bronze wire, will be given further consideration at a meeting of the committee in June, following which they may be presented to the Society through Committee E-10 on Standards.[6]

As planned by this subcommittee, the Specifications for Copper-Silicon Alloy Rods, Bars and Shapes (B 98 – 40) and for Copper-Silicon Wire for General Purposes (B 99 – 40) are being revised with respect to changes in chemical composition. This work is being done in cooperation with Subcommittee IV, inasmuch as it seems desirable to have each alloy definitely described as to nominal composition and range. The subcommittee is considering the recommendation that a new clause be inserted in the Tentative Specifications for Copper Rods, Bars, and Shapes (B 133 – 40 T) to specify the use of phosphorized copper when the copper is to be welded. Values for soft and half-hard rods are to be included in the Standard Specifications for Free-Cutting Brass Rod for Use in Screw Machines (B 16 – 29) and the specifications revised as noted earlier in this report.

A subgroup was appointed to study, in cooperation with the Frankford Arsenal, the development of quality control requirements for copper used in pressure cylinders. Samples of this material have been circulated for study, and special lots of copper have been prepared for the Arsenal in accordance with the proposed specifications. It was agreed by the committee that these specifications would not be issued by the Society, but that the committee would cooperate as fully as possible with the War Department in the development of the requirements for this material.

Subcommittee VI on Condenser Tubes (G. C. Holder, chairman) has held no meetings this year and has not found it necessary to recommend changes in any specifications under its jurisdiction.

Subcommittee VII on Copper or Deoxidized Copper Tubes (R. S. Pratt, chairman) has recommended revisions in the Tentative Specifications for Seamless Copper Tubes (B 75 – 40 T), for Seamless Copper Tubing, Bright Annealed (B 68 – 39 T), and for Copper Pipe, Standard Sizes (B 42 – 39 T), as given earlier in this report. The revision in Specifications B 75 includes the addition of requirements for methods of test and oxygen-free and deoxidized copper and also requirements for electrical conductivity. The latter were prepared in cooperation with Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors.

Early this year H. Y. Bassett found it necessary to resign as chairman and as an active participant in committee work due to a change in the work under his control. The committee accepted Mr. Bassett's resignation with regret and elected R. S. Pratt as the new chairman of the subcommittee.

Subcommittee VIII on Copper Alloy Tubes for General Use (Alan Morris, chairman) has not been active during the year inasmuch as it has not been found necessary to recommend any changes in the specifications under its jurisdiction.

Subcommittee IX on Copper-Base Alloy Forgings (J. J. Kanter, chairman), likewise, has not been active during the year and has not found it necessary to consider any further changes in the specifications under its jurisdiction.

Subcommittee X on Copper-Base Alloys for Sand Castings (G. H. Clamer, chairman) has completed seven new tentative specifications covering copper alloy sand castings as mentioned earlier in this report. Of these, two represent a revision of, and replace, two existing standard specifications, the withdrawal of which is recommended; four are new specifications; and one is a replacement of an existing tentative standard. In the Tentative Specifications for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 40 T) a revision is being recommended in the composition requirements for aluminum-bronze ingots.

Subcommittee XI on Methods of Test for Copper and Copper Alloys (A. J. Phillips, chairman) has conducted three investigations during the year, one on the mercurous-nitrate test in connection with which two papers are to be presented before the annual meeting of the Society; the second on the expansion pin test for copper and copper-alloy tubes; and the third on methods of examination and test of phosphorized and oxygen-free types of copper. The first two investigations have resulted in the preparation of proposed Tentative Methods of Mercurous Nitrate Test for Copper and Copper Alloys and a Test for Expansion (Pin Test) of Copper and Copper Alloy Tubing as mentioned earlier in this report. Upon completion of the third investigation, it is anticipated that a procedure will be recommended to those subcommittees interested in including this form of test in their specifications. The studies made in connection with the development of the mercurous nitrate test are described in two papers appearing as appendices[7] to this report by H. Rosenthal and A. L. Jamieson on "Mercury Cracking Test, Procedure and Control," and by Harry P. Croft on "Influence of External and Stresses on Tendency of Brass Wires to Stress-Corrosion Crack, as Indicated by the Mercurous Nitrate Test."[8]

Subcommittee XII on Publication of General Information (C. S. Cole, chairman) is making another survey on the possibility of publishing all of the copper-base alloy specifications in a separate volume. This subcommittee has also prepared a special clause which is to be recommended for inclusion in all of the specifications under the jurisdiction of Committee B-5. This clause will indicate the type of information or instruction that the purchaser of a material under an A.S.T.M. specification should include in the purchase order. The matter of revision of Campbell's List of Alloys is under consideration. It may be undertaken provided Committee B-5 can obtain sufficient cooperation from the other interested committees.

Subcommittee XIII on Tolerances (R. G. Thompson, chairman) was organized during the early part of this year. The work of this committee is proceeding, and it is anticipated that within the year numerous actions affecting A.S.T.M. specifications will be recommended to the Society. This committee was organized primarily for the purpose of setting up a means of active cooperation with the Copper and Brass Research Association in the development of tolerances for copper and copper-alloys in behalf of the consumer interest. The personnel of the subcommittee is restricted to consumer representation. Representatives of the various Federal agencies and a representative group of consumers are cooperating in this work. Revisions in tolerances, to be recommended to the various specification writing subcommittees of Committee B-5, will probably result from the work of this subcommittee.

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[7] See p. 897.
[8] See p. 905.

This report has been submitted to letter ballot of the committee which consists of 88 voting members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. H. GREENALL,
Chairman.

C. H. DAVIS,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee B-5 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Phosphor Bronze Rods, Bars, and Shapes (B 139 – 41 T),
Phosphor Bronze Wire (B 159 – 41 T), and

Revision of:

Tentative Specifications for Beryllium-Copper Alloy Bars, Rods, Sheet, Strip, and Wire (B 120 – 41 T).

These recommendations were accepted by Committee E-10 on August 25, 1941, assigned the A.S.T.M. serial designations indicated above, and the new and revised tentative standards appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 427, 435, and 475.

On November 8, 1941, Committee E-10 accepted a tentative revision proposed by Committee B-5 in the Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 – 40) which appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 595.

On November 28, 1941, Committee E-10, on the recommendation of Committee B-5, accepted new Tentative Specifications for Aluminum Bronze Sheet and Strip (B 169 – 41 T) which are published in the special compilation of "A.S.T.M. Standards on Copper and Copper Alloys," 1941, p. 305, and are also available as a separate reprint.

REPORT OF COMMITTEE B-6

ON

DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: one in Atlantic City, N. J., on June 27, 1940, and one in Washington, D. C., on March 4, 1941, with an attendance of 29 and 25, respectively. Certain of the subcommittees also held other meetings at various times during the year.

A balance of $138.42 is reported by Society headquarters in the fund of Committee B-6 as of December 31, 1940.

The committee has undertaken to raise $2000 to defray the expense of conducting atmospheric exposure tests on specimens of zinc, aluminum, and magnesium alloys. It is estimated that $2000 will cover the payment of an existing bill for $416.30, incurred in the construction of exposure racks, and will pay the packing and shipping charges when the present exposure tests have been completed. The committee appointed to solicit contributions from the producers and consumers of die castings to finance the present research program consists of G. O. Hiers, chairman, W. M. Peirce, A. E. Keskulla, J. C. Fox, and C. R. Ince.

In order to facilitate the creation of a new committee to study methods of test and specifications for plated metal coatings, this committee expressed its willingness to relinquish that part of its scope relating to specifications for plated metal coatings and their methods of test and sampling. Similar action by other affected committees resulted in the organization of Committee B-8 on Electrodeposited Metallic Coatings.

In order that all of the die casting activities of the Society will properly come under the jurisdiction of this committee, the scope of Committee B-6 was revised to read as follows:

> *Scope.*—Aluminum, copper, iron, lead, magnesium, tin and zinc-base die-casting alloys, or any other alloys cast in molds or dies under pressures other than gravity and usually substantially greater than atmospheric pressure. Formulation of specifications and methods of test. Studies of finishing properties and corrosion resistance of die castings. Also studies of the art of die casting and the material needs for die-casting machines and dies.

Committee B-6 has under consideration the possible formation of a new subcommittee to study brass die castings. This subject has not created much interest in the past because of the inadequate life of the die steels. The search for more suitable steels has been attempted unsuccessfully both by laboratories and, several years ago, by the former Subcommittee XXIII on Die Steels, of Committee A-1 on Steel. It seems likely that this will be an obstacle to the increase in the application of brass die castings in the near future. However, it was deemed advisable to investigate thoroughly whether there was sufficient interest in brass die casting to warrant the formation of a new subcommittee either to undertake active research work or merely to write specifications for the alloys now being die cast. The following committee was appointed to investigate this subject and to report to Committee B-6 at its next meeting: J. C. Fox, chairman, J. R. Freeman, Jr., and Sam Tour.

A number of additions and changes in the membership of the committee have been approved during the year. At the present time the committee consists of 82 members, 65 of whom are voting members; 26 are classified as producers, 20 as consumers, and 19 as general interest members.

I. Revision of Tentative Standard

The committee recommends that the Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 38 T)[1] be revised as follows and continued as tentative:

Table I.—In the table of requirements for chemical composition of alloy No. XXV, change the minimum magnesium limit from "0.02" to "0.03" per cent and change the maximum allowable tin limit from "0.002" to "0.005" per cent.

This recommendation has been submitted to letter ballot of the committee which consists of 65 voting members; 43 members returned their ballots, of whom 32 have voted affirmatively, 4 negatively, and 7 members marked their ballots "not voting." The classified vote on this recommendation was as follows: Affirmative: 9 producers, 14 consumers, and 9 general interest; negative: 2 producers and 2 consumers.

II. Tentative Standards Continued as Tentative

The committee recommends that the following tentative specifications be continued as tentative without revision:

Tentative Specifications for:

Aluminum-Base Alloy Die Castings (B 85 – 39 T),

Magnesium-Base Alloy Die Castings (B 94 – 40 T), and

Lead- and Tin-Base Alloy Die Castings (B 102 – 39 T).

Activities of Subcommittees

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. E. Thum, chairman) has under consideration with Subcommittee II on Aluminum Alloy Castings, of Committee B-7 on Light Metals and Alloys the study of the effects of minor alloying elements on aluminum alloys.

Subcommittee I is still planning to arrange with two producers for the die casting of test specimens of an alloy of 8 per cent magnesium, remainder aluminum, in order to study the effect of time on composition, that is, loss of magnesium and increase of iron.

Subcommittee II on Zinc-Base Die-Casting Alloys (C. T. Prendergast, chairman) has recommended a revision of the Tentative Specifications for Zinc-Base Alloy Die-Castings (B 86 – 38 T) with respect to the composition requirements for alloy No. XXV as mentioned earlier in this report.

Subcommittee III on Tin- and Lead-Base Die-Casting Alloys (G. O. Hiers, chairman) is continuing its studies on the five lead- and tin-base die-casting alloys. The creep studies on these alloys have been started, but no data will be available for at least another year.

Subcommittee IV on Physical Tests of Die Castings (R. L. Templin, chairman) has under consideration the selection or development of a test method which will expand the field and utility of die castings. The first approach to this problem by the subcommittee will be to canvass the members of Committee B-6 to determine if some producer or consumer of die castings is using or has any definite ideas on the development of such a test.

Subcommittee IV has brought to the attention of the Section on Impact Testing, of Committee E-1 on Methods of Testing, the matter of including in the

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 1153.

Tentative Methods of Impact Testing of Metallic Materials (E 23 – 34 T) the details of the unnotched specimen ordinarily used in testing die castings.[2]

This subcommittee plans to propose to Committee B-6 that it sponsor a paper to be prepared by the New Jersey Zinc Co. on the fatigue strength of zinc alloy die castings. It is hoped that the experimental work will be completed in time to present the paper at the 1942 annual meeting of the Society.

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman) is continuing its studies of the atmospheric corrosion tests of die-casting alloys.

The 12 aluminum-base alloys, Nos. I to XII, still on exposure are scheduled to remain there for a total of 15 yr., unless, when the time arrives, it seems advisable to extend the exposure period. The 15-yr. period will have elapsed by September, 1944. The same applies to the one zinc-base alloy, No. XXI, remaining on exposure from the original tests started in 1929.

The next tests on the three special aluminum-base alloys, Nos. IVa, Va, and V, are not scheduled until December, 1944, at which time the specimens will have been exposed for 10 yr.

The first lot of specimens to be tested from the three new zinc-base alloys, Nos. XXIa, XXIII, and XXV, and the four magnesium-base alloys, Nos. 307, 312, 313, and 314, are scheduled for August, 1944, at which time the specimens will have been exposed for 5 yr.

Subcommittee VI on Finishing Properties of Die Castings (J. C. Fox, chairman) is sponsoring a series of papers dealing with the methods of finishing the various die-cast materials since this subject is one of much importance. Most of the alloys used in the die casting process require metallic or organic coatings to protect the underlying base metal against tarnish and oxidation by the atmosphere and against corrosion by other media. The value of decorative finishes for eye appeal and for sales cannot be overstressed.

As a result of a consistent and widespread demand for the latest information on the finishing of die castings, Subcommittee VI offers this series of papers dealing with the methods of finishing zinc-, aluminum- and magnesium-base alloy die castings. Since the paper[3] on this subject, sponsored by the committee in 1936, there has been considerable change in methods of finishing, and several new types of finish have been developed. The present series of three papers describe up-to-date methods covering electroplating, chemical treatments, anodic oxidation, and organic finishes. A further paper covering the finishing of tin- and lead-base die castings is in preparation.

The subcommittee earnestly trusts that the information presented in the following three papers which appear as appendices[4] to this report will serve as a sound basis for further study of this important subject:

> "Finishes for Aluminum Die Castings," by A. E. Keskulla and Junius D. Edwards.
>
> "Finishing Magnesium Die Castings," by H. W. Schmidt, and
>
> "The Finishing of Zinc-Base Alloy Die Castings," by E. A. Anderson.

Subcommittee VII on Magnesium-Base Die-Casting Alloys (G. A. Gann, chairman) has under consideration a proposed revision of the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 – 40 T) to incorporate requirements for a new alloy to be known as No. 13X. This alloy is the same as alloy No. 13 except that the iron and nickel contents are each held under maximum values of 0.005 per cent and the maximum allowable silicon content is reduced from 0.5 to 0.3 per cent. According to the sponsor of the alloy, this restriction in allowable impurities will not in any way affect the mechanical properties but greatly increases its resistance to salt-water corrosion.

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[2] A revision of these methods in which this information has been included was issued in August, 1941, see Editorial Note, p. 482.

[3] J. C. Fox, "Finishing of Die Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 193 (1936).

[4] See pp. 931, 941, 949.

This report has been submitted to letter ballot of the committee which consists of 65 voting members; 55 members returned their ballots, of whom 50 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 on Light Metals and Alloys, Cast and Wrought, has held two meetings during the past year: at Atlantic City, N. J., on June 27, 1940, and at Washington, D. C., on March 4, 1941. The various subcommittees have held one meeting during this period.

Since the last annual meeting the committee has lost two members through death. This loss has been deeply regretted by the membership. The consolidation of the U. S. Navy Bureau of Construction and Repair and the Bureau of Engineering has also accounted for the loss of a member. Three new members have been appointed, and one member reappointed following a change in employment. The membership of the committee therefore still totals 47, of whom 22 are classified as producers, 17 as consumers, and 8 as general interest members.

Several new subcommittee appointments have likewise been made, including the appointment of representatives of Committee B-7 on several other standing committees. Mr. A. Sugar of the Monarch Aluminum Manufacturing Co. has been elected chairman of Subcommittee I to replace D. L. Colwell who resigned during the year.

In general, the activities of Committee B-7 and its subcommittees during the year have been directed toward assisting in the various production problems which have arisen because of the increased demand for light metals and their alloys. This activity is reflected in the various changes which have been proposed in specifications under their jurisdiction. Alloy composition changes, revisions in tolerances, and the addition of a new sand casting alloy developing good strength without the necessity of heat treatment are among these changes. Although some of these specifications should normally be considered for adoption as standard at this time, the uncertainty resulting from production demands has made it desirable to withhold this action for the future.

I. Revisions of Tentative Standards

The committee recommends that the following tentative specifications be revised as indicated in Appendix I and continued as tentative. These revisions are desired in order to bring these specifications in line with present commercial practices and to meet a consumer demand for products which have not previously been covered:

Tentative Specifications for:

Aluminum Ingots for Remelting (B 24 – 39 T)
Aluminum Sheet and Plate (B 25 – 40 T)
Aluminum-Base Alloy Sand Castings (B 26 – 37 T)
Aluminum for Use in Iron and Steel Manufacture (B 37 – 39 T)
Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 37 T)
Aluminum Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 – 40 T)
Aluminum-Manganese Alloy Sheet and Plate (B 79 – 40 T)
Magnesium-Base Alloy Sand Castings (B 80 – 40 T)
Aluminum Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 40 T)

Magnesium-Base Alloy Sheet (B 90 – 40 T)
Magnesium-Base Alloy Forgings (B 91 – 40 T)
Magnesium-Base Alloys in Ingot Form for Sand Castings and Die Castings (B 93 – 40 T)
Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 40 T)
Aluminum-Base Alloy Permanent Mold Castings (B 108 – 38 T)
Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 40 T)
Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 – 38 T)

II. REVISION OF STANDARD

The committee recommends for immediate adoption, a revision in the Standard Specifications for Magnesium Ingot and Stick for Remelting (B 92 – 39) in order to bring the composition requirements into line with commercial practices, as indicated in Appendix I, and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society.

III. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

In accordance with the regulations governing tentative standards, the committee has also reviewed the Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Die Castings (B 125 – 39 T) and the Tentative Method of Test for Dielectric Strength of Anodized Aluminum (B 110 – 39 T) which have stood two years without revision. It is recommended that these two tentative standards be continued as tentative without revision, the first to await the adoption as standard by Committee B-6 on Die-Cast Metals and Alloys of the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 39 T), and the second to await further comments from consumers.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 47 members; 31 members having returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Item	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Aluminum Ingots for Remelting (B 24 – 39 T)	20	0	11
Spec. for Aluminum Sheet and Plate (B 25 – 40 T)	23	0	8
Spec. for Aluminum-Base Alloy Sand Castings (B 26 – 37 T)	27	0	4
Spec. for Aluminum for Use in Iron and Steel Manufacture (B 37 – 39 T)	17	0	14
Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 37 T)	29	0	2
Spec. for Aluminum Alloy (Duralumin) Sheet and Plate (Aluminum-Copper-Magnesium-Manganese) (B 78 – 40 T)	22	0	9
Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 40 T)	22	0	9
Spec. for Magnesium-Base Alloy Sand Castings (B 80 – 40 T)	23	1	7
Spec. for Aluminum Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 40 T)	21	0	10
Spec. for Magnesium-Base Alloy Sheet (B 90 – 40 T)	23	0	8
Spec. for Magnesium-Base Alloy Forgings (B 91 – 40 T)	23	0	8
Spec. for Magnesium-Base Alloys in Ingot Form for Sand Castings and Die Castings (B 93 – 40 T)	25	0	6
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 40 T)	22	0	9
Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 38 T)	28	0	3
Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 40 T)	21	0	10
Spec. for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 – 38 T)	28	0	3
II. REVISION OF STANDARD			
Spec. for Magnesium Ingot and Stick for Remelting (B 92 – 39), immediate adoption	26	0	5

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (A. Sugar, chairman).—This subcommittee, after review of the various specifications under its jurisdiction, has recommended revisions of the Tentative Specifications for Aluminum Ingots for Remelting (B 24 – 39 T), Aluminum for Use in Iron and Steel Manufacture (B 37 – 39 T), and Aluminum-Base Alloys in Ingot Form for Sand Castings and Permanent Mold Castings (B 58 – 37 T and B 112 – 38 T). The details of these revisions are given in Appendix I. Of principal interest has been the addition of a 99.2 per cent grade aluminum for remelting purposes and a modification of the zinc content in the 94.0 per cent grade for use in iron and steel.

Subcommittee II on Aluminum Alloy Castings (C. H. Greenall, chairman).—Revisions in the Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 37 T) and for Permanent Mold Castings (B 108 – 38 T) have been prepared by this subcommittee. Principal among these recommended revisions is the addition of a new sand casting alloy with good mechanical properties in the as-cast condition. A table of general information and typical yield strengths for the sand casting alloys has also been prepared for addition to these specifications. The details of these proposed revisions are given in Appendix I. There is included in Appendix II to this report information and data, prepared by Mr. D. Basch, covering the new aluminum-zinc-magnesium sand-casting alloy "O" being recommended for inclusion in Specifications B 26 – 37 T.

The study of the effect of minor elements on the characteristics of aluminum alloy castings is still under consideration by a subgroup. Progress has been slow, however, due to the press of other business. It is hoped that something definite can be reported at the next annual meeting.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (P. V. Faragher, chairman).—In order to bring the requirements of the various specifications for aluminum wrought shapes in line with present commercial practices, revisions in certain tolerances have been found necessary. Tolerance revisions in connection with the Tentative Specifications for Aluminum Sheet and Plate (B 25 – 40 T), Aluminum Alloy (Duralumin) Sheet and Plate (B 78 – 40 T), Aluminum-Manganese Alloy Sheet and Plate (B 79 – 40 T), Aluminum Alloy (Duralumin) Bars, Rods, Wire, and Shapes (B 89 – 40 T), and Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 40 T) have been recommended. The details of these recommended tolerance changes and a minor change in composition are given in Appendix I.

This subcommittee has also set up a subgroup to study the desirability of consolidating the wrought alloy specifications under their jurisdiction.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (J. A. Gann, chairman).—This subcommittee has recommended changes in six specifications under its jurisdiction. In the case of the present Standard Specifications for Magnesium Ingot and Stick for Remelting (B 92 – 39), a minor change in composition is proposed for immediate adoption. In the case of the Tentative Specifications for Magnesium-Base Alloy Sand Castings (B 80 – 40 T), Sheet (B 90 – 40 T), Forgings (B 91 – 40 T), Ingots for Sand Castings and Die Castings (B 93 – 40 T), and Bars, Rods, and Shapes (B 107 – 40 T), additional high-purity grades of certain of the alloys have been proposed. The detailed changes are given in Appendix I.

Although these high-purity alloys offer no better mechanical properties than the present commercial purity alloys, they do provide an improved resistance to salt water corrosion, which is in demand for certain applications. The limited sources of supply for the high-purity alloys and the premium prices have been pointed out. Data showing the loss in weight after alternate immersion in a 3 per cent sodium chloride solution of all the magnesium alloys now covered by A.S.T.M. specifications and of the proposed new high-purity alloys are given in Table II. The data indicate the fluctuations that may be expected in this type of test. The parenthetical values given for the present normal grade of alloy represent the typical corrosion rate, or the maximum in the frequency distribution curve. Corresponding values for the high-purity grade alloys are not included inasmuch as the individual test data are in good agreement. The data in Table II indicate that these high-purity alloys should be recommended when consistent maximum resistance to salt water is required.

TABLE II.—DATA ON CORROSION OF MAGNESIUM ALLOYS IN ALTERNATE IMMERSION TESTS IN SODIUM CHLORIDE SOLUTION (3 PER CENT).

Alloy	Condition	Loss in Weight after Alternate Immersion in 3 per cent NaCl, mg. per sq. cm. per day	
		Normal Grade[a]	High-Purity Grade[a]
SAND CAST ALLOYS (A.S.T.M. DESIGNATION: B 80 - 40 T)[b]			
No. 2	Heat treated and aged	1.0 to 100 (26.0)[c]	....
No. 3	Heat treated and aged	1.0 to 100 (26.0)	....
No. 4	As cast	0.2 to 20 (1.5)	0.2 to 0.5
	Heat treated	0.5 to 50 (5.0)	0.2 to 0.5
	Heat treated and aged	0.2 to 30 (2.0)	0.2 to 0.5
No. 11	As cast	0.2 to 2.0 (0.4)	....
No. 14	Heat treated and aged	0.5 to 30 (6.0)	....
No. 17	As cast	0.2 to 25 (2.0)	0.2 to 0.5
	Heat treated	0.5 to 60 (7.0)	0.2 to 0.5
	Heat treated and aged	0.2 to 30 (2.5)	0.2 to 0.5
DIE-CAST ALLOYS (A.S.T.M. DESIGNATION: B 94 - 40 T)[b]			
No. 12	As cast	0.5 to 10 (2.0)	....
No. 13	As cast	0.3 to 10 (2.0)	0.1 to 0.5
FORGED AND EXTRUDED ALLOYS (A.S.T.M. DESIGNATIONS: B 91 - 40 T AND B 107 - 40 T)[b]			
No. 8	As forged and extruded	0.7 to 10 (5.0)	0.1 to 1.0
No. 9	As forged and extruded	0.7 to 15 (5.0)	0.1 to 1.0
No. 11	As forged and extruded	0.2 to 4.0 (0.6)	....
No. 15	As forged and extruded	0.5 to 10 (2.0)	0.2 to 1.0
No. 16	As forged and extruded	0.7 to 10 (5.0)	....
No. 18	As forged and extruded	0.7 to 10 (3.0)	0.1 to 1.0
SHEET ALLOYS (A.S.T.M. DESIGNATION: B 90 - 40 T)[b]			
No. 7	As rolled	1.0 to 100 (15.0)	....
No. 8	As rolled	0.7 to 10 (5.0)	0.1 to 1.0
No. 11	As rolled	0.2 to 4.0 (0.6)	....
No. 18	As rolled	0.7 to 10 (3.0)	0.1 to 1.0

[a] The normal grade alloys now used in industry are designated by the A.S.T.M. as alloys Nos. 2, 3, 4, 8, etc. The corresponding high-purity grade alloys are designated as alloys Nos. 4X, 8X, etc.

[b] 1940 Supplement to Book of A.S.T.M. Standards, Part I, pp. 343, 446, 351, 347, and 355.

[c] The parenthetical values in this column represent the typical corrosion rate or the maximum in the frequency distribution curve.

Subcommittee V on Testing Light Alloys (R. L. Templin, chairman).—This subcommittee has given consideration to the determination of the yield strength of the light alloys by means of the divider method. Such data as has been presented on this subject has indicated that the method is not sufficiently accurate for control purposes in connection with tension testing. A memorandum on this method by H. W. Schmidt, entitled, "The Divider Method in Tension Testing of Magnesium-Base Alloys" appears in Appendix III.

This subcommittee is also studying the revised Tentative Methods of Tension Testing of Metallic Materials (E 8 - 40 T), particularly as they apply to the testing of light alloys. In this connection certain discrepancies in the illustrations of the $\frac{1}{2}$ in. diameter test specimens appearing in the various B-7 specifications are being reviewed for the purpose of establishing uniformity in the dimensional requirements for these specimens.

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (J. D. Edwards, chairman).—A review by this subcommittee of the various methods under its jurisdiction has indicated that no revisions are necessary at this time. The Tentative Methods of Test for Dielectric Strength of Anodized Aluminum (B 110 - 39 T), Test for Sealing of Anodically Coated Aluminum (B 136 - 40 T), and Test for Weight of Coating on Anodically Coated Aluminum (B 137 - 40 T) are being continued as tentative.

Progress has been reported in connection with the program designed to compare the performance of anodic coatings in the salt spray test (B 117 - 39 T) with their behavior under atmospheric exposure. The subgroup following this investigation has prepared fourteen groups of nine samples each, comprising coatings made on two different aluminum alloys and with variations in the electrolyte used for coating, variations in the thickness of coating, and in the method of sealing. Six organizations have agreed to cooperate in the testing of these samples and tests should be started shortly.

Subcommittee VII on General Information and Publicity (E. H. Dix, Jr., chairman).—As a result of a survey made by this subcommittee and reported to Committee B-7 at its meeting in June, 1940, it was recommended that the Report on Service Characteristics of the Light Metals and Their Alloys, published in 1934,[1] be declared obsolete and that no revision be undertaken. Since the preparation of this report constituted the principal function of this subcommittee, it has since been discharged.

Special Subcommittee on Classification and Designation of Aluminum Alloys (I. V. Williams, chairman).—This subcommittee, organized last year to develop an alloy nomenclature system, reports progress and as a result of considerable constructive discussion at the last meeting, will probably present its recommendations shortly. The committee plans to expand its tentative aluminum system to the magnesium-base alloys and also to have it considered by Committee B-6 on Die-Cast Metals and Alloys in connection with the aluminum and magnesium die-casting alloys.

This report has been submitted to letter ballot of the committee which consists of 47 members; 31 members returned their ballots, of whom 29 have volted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SAM TOUR,
Chairman.

H. J. ROWE,
Secretary.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 277 (1934); also issued as separate publication.

APPENDIX I

PROPOSED REVISIONS IN STANDARDS FOR LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given proposed revisions in certain specifications covering light metals and alloys, cast and wrought, which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the specifications appear in their present form.

Tentative Specifications for Aluminum Ingots for Remelting (B 24 – 39 T):[1]

Section 1.—Change the second sentence to read as follows by the addition of the italicized word and figures and the omission of the word in brackets:

> [Three] *Four* grades are specified and are designated grades 99.5, *99.2*, 99.0, and 98.0.

Table I.—In the table of chemical requirements, change the iron content for grade 99.5 from "0.5 max. per cent" to read "0.4 max. per cent," and add the following requirements for a new grade, designated 99.2:

	Grade 99.2
Aluminum, min., per cent by difference	99.2
Other elements, max., per cent:	
Copper	0.1
Iron	0.4
Silicon	0.4
Magnesium	0.03
Manganese	0.03
Zinc	0.03
Titanium	0.03
Nickel	0.03
Total other impurities, max., per cent	0.1[a, b]

[a] None of the elements under the heading of total other impurities shall exceed 0.03 per cent maximum.

[b] When this material is specified for use in making zinc-base die-casting alloys, the lead, cadmium, or tin shall not exceed a maximum of 0.01 per cent for each.

Tentative Specifications for Aluminum Sheet and Plate (B 25 – 40 T):[2]

Table II.—In the section of this table covering permissible variations in thickness of flat sheet, delete the first column of tolerances headed "18 in. and Under in Width" and change the heading of the second column to read "36 in. and Under in Width."

Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 37 T):[1]

Section 1.—Change the second sentence to read as follows by the addition of the italicized word and letter and the omission of the word in brackets:

> [Twelve] *Thirteen* alloys are specified and are designated alloys C, CC, F, G, GG, H, J, JJ, K, L, M, N, and *O* (see Explanatory Notes).

Table I.—Change the magnesium requirement for alloy GG from "0.3 max. per cent" to read "0.10 max. per cent."

Add to this table the following chemical requirements for a new alloy, designated alloy O:

	Alloy O
Aluminum, per cent	remainder
Copper, per cent	0.2
Iron, per cent	1.0
Silicon, per cent	0.6
Magnesium, per cent	0.5 ± 0.1
Manganese, per cent	...
Zinc, per cent	$5.0\left\{\begin{matrix}+0.7\\-0.2\end{matrix}\right.$
Titanium, per cent	0.2 ± 0.1
Chromium, per cent	0.5 ± 0.1
Total other impurities, per cent	0.05 each

NOTE.—Where single limits are shown, these indicate the maximum amounts permitted.

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[1] 1939 Book of A.S.T.M. Standards, Part I.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part I.

Table II.—Change the tensile requirements for alloy F from their present form to read as follows by the addition of the italicized words and figures and the omission of those in brackets, and also add the following requirements for a new alloy, designated alloy O:

Alloy	Condition	Tensile Strength, min., psi. (to the nearest 1000 psi.)	Elongation in 2 in., min., per cent (to the nearest 1 per cent)
F	[As cast]	[19 000]	[not required]
	Heat-treatment No. 1	*23 000*	*6*
	Heat-treatment No. 2	*30 000*	*6*
O	Aged	30 000	3

From Table II delete the requirements for alloy N, Heat-Treatment No. 1.

Explanatory Notes.—Under Alloy F, change the specific gravity from "about 2.90" to read "approximately 2.85."

Under Alloy G, change the specific gravity from "about 2.78" to read "approximately 2.77."

Under Alloy GG, add the following sentence at the beginning of this paragraph: "The specific gravity of alloy GG is approximately 2.77."

Under Alloy H, change the specific gravity from "about 2.80" to read "approximately 2.73."

Under Alloy J, change the specific gravity from "about 2.69" to read "approximately 2.66."

Under Alloy JJ, add the following sentence at the beginning of this paragraph: "The specific gravity of alloy JJ is approximately 2.66."

Under Alloy K, change the specific gravity from "about 2.68" to read "approximately 2.65."

Under Alloy N, change the fourth sentence from its present form to read as follows: "Heat-treatment No. 2, consisting of a solution and precipitation treatment, provides the optimum combination of strength and ductility for this alloy."

Add a new paragraph to read as follows at the end of the present notes:

Alloy O.—This alloy has an approximate specific gravity of 2.80. It is a general-purpose alloy having high physical properties which are obtained by either room temperature aging of several weeks duration or by artificial aging. Solution heat-treatment is not required. Within the first two weeks after casting, the alloy has high ductility, permitting severe cold deformation.

Make the present explanatory notes "Note 1," and add the following as Note 2:

NOTE 2.—The yield strength of aluminum-base alloys is defined as the stress at which the stress-strain curve deviates 0.2 per cent from the modulus line. It may be determined by the "Offset Method" or the "Extension Under Load Method" (the latter is often referred to as the "Approximate Method Without the Stress-Strain Diagram") as described in the Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials. The following data give typical yield strength values for the sand-cast aluminum alloys:

Alloy	Condition	Typical Yield Strength (0.2 per cent offset), psi.
C and CC	As cast	14 000
F	Heat-treatment No. 1	20 000
	Heat-treatment No. 2	30 000
G	Heat-treatment No. 1	16 000
	Heat-treatment No. 2	22 000
	Heat-treatment No. 3	31 000
GG	Heat-treated	31 000
H	Heat-treated	32 000
J and JJ	As cast	8 000
K	As cast	11 000
L	As cast	12 000
M	Heat-treatment No. 1	16 000
	Heat-treatment No. 2	22 000
N	Heat-treatment No. 2	25 000
	Heat-treatment No. 3	23 000
O	Aged four weeks at room temperature or artificially aged	25 000

Appendix.—Add the accompanying Table I covering supplementary data as an Appendix to the specifications.

Tentative Specifications for Aluminum for Use in Iron and Steel Manufacture (B 37 – 39 T):[1]

Section 1—Change the second sentence to read as follows by the addition of the italicized word and figures and the omission of the word in brackets:

> [Four] *Five* grades are specified and are designated grades *90.0*, 94.0, 96.0, 98.0, and 99.0.

TABLE I.—SUPPLEMENTARY DATA.

Note.—The data in this table are approximate and are supplied for general information only.

TABLE OF SUPPLEMENTARY DATA.

Alloy	Approximate Specific Gravity	Corrosion Resistance	Casting Properties	Response to Heat Treatment	Principal Uses	Machinability
C..........	2.85	Fair	Very good	No	Automotive crankcases, oil pans, transmission cases, miscellaneous general-purpose castings	Excellent
CC..........	2.83	Fair	Very good	No	General castings, patterns	Excellent
F..........	2.85	Fair	Good	Yes	Air-cooled cylinder heads, pistons, and castings requiring good strength and hardness at elevated temperatures	Excellent
G..........	2.77	Good	Good	Yes	General, where high strength and ductility are required	Very good
GG..........	2.77	Good	Good	Yes	General, where high yield strength and hardness are required	Very good
H..........	2.73	Good	Fair	Yes	Air-cooled cylinder heads, pistons, and castings requiring strength and hardness at elevated temperatures	Very good
J..........	2.66	Good	Excellent	No	General castings requiring pressure tightness	Fair
JJ..........	2.66	Very good	Excellent	No	Cooking utensils, marine fittings, food handling equipment, and ornamental parts requiring good resistance to corrosion	Fair
K..........	2.65	Very good	Excellent	No[a]	General, where strength and ductility in excess of that provided by other as-cast alloys is required	Fair
L..........	2.63	Excellent	Fair	No	Cooking utensils, food handling equipment, marine fittings, and other castings requiring maximum resistance to corrosion	Very good
M..........	2.65	Very good	Excellent	Yes	General, where high strength and corrosion resistance are required	Good
N..........	2.68	Good	Excellent	Yes	Aircraft engine crankcases, oil pans, liquid cooled cylinder heads, and other intricate high strength castings	Good
O..........	2.80	Good	Good	[b]	General, where high strength and ductility are required	Excellent

[a] Properties improved by modification process.

[b] See Explanatory Note 1.

Table I.—Change the requirement for zinc content of the 94.0 grade from "0.5 max. per cent" to read "1.0 max. per cent."

Add to this table the following chemical requirments for a new grade designated grade 90.0:

	Grade 90.0
Aluminum, min., per cent by difference	90.0
Other elements, max., per cent:	
Copper	4.0
Iron	10.0
Silicon	10.0
Magnesium	5.0
Manganese	1.5
Zinc	2.0
Nickel	0.5
Chromium	0.2
Tin plus lead	0.5
Other impurities, max., per cent	0.5

Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 37 T):[1]

Table I.—Change the requirement for magnesium content of alloy GG from "0.3 max. per cent" to read "0.10 max. per cent."

Tentative Specifications for Aluminum Alloy (Duralumin) Sheet and Plate (Aluminum - Copper - Magnesium - Manganese) (B 78 – 40 T):[2]

Section 3.—In the tabulation of chemical requirements, change the magnesium content from "0.2 to 0.75 per cent" to read "0.2 to 0.8 per cent" and the silicon content from "0.75 max. per cent" to read "0.8 max. per cent."

TABLE II.—PERMISSIBLE VARIATIONS IN THICKNESS, PLUS OR MINUS, IN.

Sheet Thickness, in.	36 in. and under in Width	Over 36 to 42 in. in Width	Over 42 to 48 in. in Width	Over 48 to 54 in. in Width	Over 54 to 60 in. in Width	Over 60 to 66 in. in Width
0.249 to 0.126	0.04T[a]	0.05T[a]	0.05T[a]	0.05T[a]	0.06T[a]	0.08T[a]
0.125 to 0.092	0.0045	0.005	0.005	0.005	0.007	0.010
0.091 to 0.065	0.003	0.004	0.004	0.005	0.006	0.008
0.064 to 0.052	0.003	0.004	0.004	0.005	0.006	0.006
0.051 to 0.041	0.003	0.004	0.004	0.005	0.006	0.006
0.040 to 0.033	0.0025	0.003	0.003	0.004	0.005	....
0.032 to 0.026	0.002	0.0025	0.0025	....	....	....
0.025 to 0.019	0.002	0.0025	....	....	....	....
0.018 to 0.015	0.002	....	....	....	....	....
0.014 to 0.010	0.0015	....	....	....	....	....

Sheet Thickness, in.	Over 66 to 72 in. in Width	Over 72 to 78 in. in Width	Over 78 to 84 in. in Width	Over 84 to 90 in. in Width	Over 90 to 96 in. in Width	Over 96 to 102 in. in Width
0.249 to 0.126	0.10T[a]	0.10T[a]	0.11T[a]	0.11T[a]	0.12T[a]	0.14T[a]
0.125 to 0.092	0.012	0.013	0.014	0.016	0.018	0.020
0.091 to 0.065	0.010	0.010	0.011	0.012	....	....
0.064 to 0.052	0.007	0.008	0.009	....	....	....
0.051 to 0.041	0.007	....	....	....	....	....

STRIP

Strip Thickness, in.	18 in. and under in Width	Over 18 to 24 in. in. Width
0.064 to 0.041	0.0025	0.003
0.040 to 0.026	0.002	0.0025
0.025 to 0.017	0.0015	0.002
0.016 to 0.012	0.0015	0.0015

[a] T = the nominal thickness.

Table I.—Delete the physical requirements for the temper "Rolled After Heat-Treatment."

Table II.—In the section of this table referring to sheet, change the permissible variations in thickness from their present form to read as prescribed in the accompanying Table II.

Tentative Specifications for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 40 T):[2]

Table II.—In the section of this table covering permissible variations in thickness of flat sheet, delete the first column of tolerances headed "18 in. and Under in Width" and change the heading of the second column to read "36 in. and Under in Width."

Tentative Specifications for Magnesium-Base Alloy Sand Castings (B 80 – 40 T):[2]

Section 1.—In the second sentence change the phrase "Six alloys" to read "Six types of alloys."

Table I.—Change the requirement for silicon content of alloy Nos. 2, 3, 4, 14, and 17 from "0.5 max. per cent" to read "0.3 max. per cent."

Add to this table the following chemical requirements for two high-purity alloys, designated Nos. 4X and 17X:

	Alloy No. 4X	Alloy No. 17X
Magnesium, per cent	remainder	remainder
Aluminum, per cent	5.3 to 6.7	8.3 to 9.7
Manganese, min., per cent	0.15	0.10
Zinc, per cent	2.5 to 3.5	1.6 to 2.4
Silicon, max., per cent	0.3	0.3
Copper, max., per cent	0.05	0.05
Nickel, max., per cent	0.005	0.005
Iron, max., per cent	0.005	0.005
Other impurities, max., per cent	0.3	0.3

Table II.—Change the present tensile requirements for alloy Nos. 4 and 17 to read as prescribed in the accompanying Table III by the addition of the italicized figures and the omission of those in brackets.

Explanatory Note 1.—Under alloy No. 17, add a second paragraph to read as follows:

> Alloy No. 17 in the heat-treated condition ages more rapidly than alloy No. 4 in the heat-treated condition. Under service conditions where the castings attain a temperature of 200 F. or higher, the castings of alloy No. 17, heat-treated, will gradually change to the heat-treated and aged condition.

TABLE III.—TENSILE REQUIREMENTS.

Alloy	Condition	Tensile Strength, min., psi.	Yield Strength (0.2 per cent offset), min., psi.	Elongation in 2 in., min., per cent
No. 4 *and 4X*	As cast	24 000	not required	4
	Heat-treated	[30 000] *32 000*	not required	[6] *7*
	Heat-treated and aged	[32 000] *34 000*	16 000	[2] *3*
No. 17 *and 17X*	As cast	20 000	not required	not required
	Heat-treated	[30 000] *32 000*	not required	6
	Heat-treated and aged	[32 000] *34 000*	18 000	1

At the end of Note 1 add a new paragraph to read as follows:

> *Alloy Nos. 4X and 17X.*—These alloys are the same as alloy Nos. 4 and 17, respectively, except that the iron and nickel contents are held to low values in order to obtain maximum resistance to salt-water (NaCl) corrosion as required for military aircraft and seaboard exposures. Resistance to normal atmospheric corrosion is not materially increased. Mechanical properties are the same as for the normal grades of alloys. These high-purity alloys are available from limited sources of supply and at a premium over the normal grades of alloys.

Table III.—In the first column insert "and 4X" after alloy No. 4, and add "and 17X" after alloy No. 17.

Tentative Specifications for Aluminum Alloy (Duralumin) Bars, Rods, Wire, and Shapes (Aluminum-Copper-Magnesium-Manganese) (B 89 – 40 T):[2]

Section 3.—In the tabulation of chemical requirements, change the magnesium content from "0.2 to 0.75 per cent" to read "0.2 to 0.8 per cent" and the silicon content from "0.75 max. per cent" to read "0.8 max. per cent."

Table III.—Delete the permissible variation "plus or minus 0.018 in." for sizes 3.001 in. and over of extruded rounds, squares, rectangles, hexagons, octagons, bars, and rods, replacing it with a reference to the following footnote to be added to the table:

> As agreed upon by the manufacturer and the purchaser.

Tentative Specifications for Magnesium-Base Alloy Sheet (B 90 – 40 T):[2]

Section 1.—Change the second sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> [Three] *Four types of* alloys are specified and are designated alloy Nos. [6, 7, and 11] *7, 8, 11, and 18* (see Explanatory Note 1).

Table I.—Delete from this table the requirements for alloy No. 6 (incorrectly designated No. 9 in present printing).

Change the requirement for silicon content of alloy No. 7 from "0.5 max. per cent" to read "0.3 max. per cent."

Add to this table the chemical requirements, prescribed in the accompanying Table IV, for four new alloys, designated Nos. 8, 8X, 18, and 18X:

Table II.—Change from its present form to read as prescribed in the accompanying Table V.

Explanatory Note 1.—Delete the first

and second paragraphs referring to alloy Nos. 6 and 7, and add the following new paragraphs:

Alloy No. 7.—This alloy has a specific gravity of approximately 1.79. It is characterized by good physical properties. The copper and nickel contents should be kept low to minimize corrosion.

Alloy No. 8.—This alloy has a specific gravity of approximately 1.80. Its resistance to salt-water (NaCl) corrosion is superior to alloy No. 7 and its physical properties are about equal to those of alloy No. 7. The copper and nickel contents should be kept low to minimize corrosion.

Alloy No. 18.—This alloy has a specific gravity of approximately 1.77. Its rolling characteristics are better than those of the other alloys. It is the alloy generally used where moderate strength and good stability are required. The copper and nickel contents should be kept low to minimize corrosion.

TABLE IV.—CHEMICAL REQUIREMENTS.

Alloy	Magnesium, per cent	Aluminum, per cent	Manganese, min., per cent	Zinc, per cent	Silicon, max., per cent	Copper, max., per cent	Nickel, max., per cent	Iron, max., per cent	Other Impurities, max., per cent
8	remainder	5.8 to 7.2	0.15	0.4 to 1.5	0.3	0.05	0.03	.....	0.3
8X	remainder	5.8 to 7.2	0.15	0.4 to 1.5	0.3	0.05	0.005	0.005	0.3
18	remainder	2.4 to 3.0	0.20	0.7 to 1.3	0.3	0.05	0.03	.....	0.3
18X	remainder	2.4 to 3.0	0.20	0.7 to 1.3	0.3	0.05	0.005	0.005	0.3

Alloy Nos. 8X and 18X.—These alloys are the same as alloy Nos. 8 and 18, respectively, except that the iron and nickel contents are held to low values in order to obtain maximum resistance to salt-water (NaCl) corrosion as required for military aircraft and seaboard exposures. Resistance to normal atmospheric corrosion is not materially increased. Mechanical properties are the same as for the normal grades of alloys. These high-purity alloys are available from limited sources of supply and at a premium over the normal grades of alloys.

Explanatory Note 2.—Change the table covering yield strength and unit deformation values from its present form to read as follows:

Alloy	Yield Strength (0.2 per cent offset), min., psi.	Unit Deformation, in. per in. of gage length
No. 7	28 000	0.0063
Nos. 8 and 8X	28 000	0.0063
No. 11	24 000	0.0057
Nos. 18 and 18X	26 000	0.0060

Tentative Specifications for Magnesium-Base Alloy Forgings (B 91 – 40 T):[2]

Table I.—Change the requirements for silicon content of alloys Nos. 8, 9, 15, and 16 from "0.5 max. per cent" to read "0.3 max. per cent."

Add to this table the following chemical requirements for a high-purity alloy, designated No. 18X:

	Alloy No. 18X
Magnesium, per cent	remainder
Aluminum, per cent	2.4 to 3.0
Manganese, min., per cent	0.20
Zinc, per cent	0.7 to 1.3
Silicon, max., per cent	0.3
Copper, max., per cent	0.05
Nickel, max., per cent	0.005
Iron, max., per cent	0.005
Other impurities, max., per cent	0.3

TABLE V.—TENSILE REQUIREMENTS.

Alloy	Condition	Tensile Strength, min., psi.	Yield Strength[a] (0.2 per cent offset), min., psi.	Elongation in 2 in. min., per cent
No. 7	Hard rolled (H)....	39 000	28 000	3
	Annealed (A)......	36 000	.....	10
Nos. 8 and 8X	Hard rolled (H)....	40 000	28 000	3
	Annealed (A)......	37 000	.....	8
No. 11	Hard rolled (H)....	32 000	24 000	4
	Annealed (A)......	28 000	.....	12
Nos. 18 and 18X	Hard rolled (H)....	38 000	26 000	4
	Annealed (A).....	32 000	.....	12

[a] See Explanatory Note 2.

Table II.—In the first column insert "and 18X" after alloy No. 18.

Explanatory Note 2.—Change the fourth paragraph from its present form to read as follows and transfer it to the end of Explanatory Note 2:

Alloy Nos. 8X, 9X, 15X, and 18X.—These alloys are the same as alloy Nos. 8, 9, 15, and 18, respectively, except that the iron and nickel contents are held to low values in order to obtain maximum resistance to salt-water (NaCl) corrosion as required for military aircraft and seaboard exposures. Resistance to normal atmospheric corrosion is not materially increased. Mechanical properties are the same as for the normal grades of alloys. These high-purity alloys are available from limited sources of supply and at a premium over the normal grades of alloys.

	Alloy No. 18X
Magnesium, per cent	remainder
Aluminum, per cent	2.4 to 3.0
Manganese, min., per cent	0.20
Zinc, per cent	0.7 to 1.3
Silicon, max., per cent	0.3
Copper, max., per cent	0.05
Nickel, max., per cent	0.005
Iron, max., per cent	0.005
Other impurities, max., per cent	0.3

Table II.—In the first column insert "and 18X" after alloy No. 18.

TABLE VI.—CHEMICAL REQUIREMENTS.

Alloy	Magnesium, per cent	Aluminum, per cent	Manganese, min., per cent	Zinc, per cent	Silicon, max., per cent	Copper, max., per cent	Nickel, max., per cent	Iron, max., per cent	Other Impurities, max., per cent
No. 4X	remainder	5.5 to 6.5	0.18	2.7 to 3.3	0.2	0.04	0.003	0.003	0.3
No. 13X	remainder	8.5 to 9.5	0.15	0.5 to 0.9	0.2	0.04	0.003	0.003	0.3
No. 17X	remainder	8.5 to 9.5	0.13	1.7 to 2.3	0.2	0.04	0.003	0.003	0.3

Explanatory Note 3.—In the first column of the table insert "and 18X" after alloy No. 18.

Tentative Specifications for Magnesium-Base Alloys in Ingot Form for Sand Castings and Die Castings (B 93 - 40 T):[2]

Section 1.—In the third sentence change the phrase "Eight alloys" to read "Eight types of alloys."

Table I.—Add to this table the chemical requirements, prescribed in the accompanying Table VI, for three high-purity alloys, designated Nos. 4X, 13X, and 17X:

Tentative Specifications for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 40 T):[2]

Table I.—Change the requirements for silicon content of alloys Nos. 8, 9, and 15 from "0.5 max. per cent" to read "0.3 max. per cent."

Add to this table the following chemical requirements for a high-purity alloy, designated No. 18X:

Section 9.—Change the table covering permissible variations from its present form to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

Material	Diameter or Distance Across Flats, in.	Permissible Variations, plus or minus, in.
Round, Square Hexagonal, and Rectangular Bars	0.500 and under	0.007
	0.501 to 1.000	0.010
	1.001 to 2.000	0.012
	2.001 to 3.000	0.015
	[3.001 and over	0.018]
	3.001 to 4.000	*0.018*
	4.001 to 5.000	*0.020*
	5.001 to 6.000	*0.022*
	6.001 to 7.000	*0.025*
	7.001 to 8.000	*0.030*
	8.001 to 9.000	*0.035*
	9.001 to 10.000	*0.040*
	10.001 to 11.000	*0.045*
	11.001 to 12.000	*0.050*
Structural Shapes	0.125 and under	0.010
	0.126 to 0.500	0.015
	0.501 to 1.000	0.020
	1.001 to 2.000	0.025
	2.001 to 3.000	0.030
	3.001 to 4.000	0.035
	4.001 to 5.000	[0.045] *0.040*
	[5.001 and over	0.060]
	5.001 to 6.000	*0.045*
	6.001 to 7.000	*0.050*
	7.001 to 8.000	*0.055*
	8.001 to 9.000	*0.060*
	9.001 to 10.000	*0.065*
	10.001 to 11.000	*0.070*
	11.001 to 12.000	*0.080*

Explanatory Note 1.—Change the fifth paragraph from its present form to read as follows and transfer it to the end of Explanatory Note 1:

Alloy Nos. 8X, 9X, 15X, and 18X.—These alloys are the same as alloy Nos. 8, 9, 15, and 18, respectively, except that the iron and nickel contents are held to low values in order to obtain maximum resistance to salt-water (NaCl) corrosion as required for military aircraft and seaboard exposures. Resistance to normal atmospheric corrosion is not materially increased. Mechanical properties are the same as for the normal grades of alloys. These high-purity alloys are available from limited sources of supply and at a premium over the normal grades of alloys.

Table III.—In the first column insert "and 18X" after alloy No. 18; also make an editorial correction in the values for yield strength and unit deformation of alloy No. 11 by changing these data from "23,000 and 0.0055" to read "20,000 and 0.0051," respectively.

TABLE VII.—PERMISSIBLE VARIATIONS IN THICKNESS OF FLAT SHEET, PLUS OR MINUS, IN.

Thickness, in.	36 in. and under in Width	Over 36 to 42 in. in Width	Over 42 to 48 in. in Width	Over 48 to 54 in. in Width	Over 54 to 60 in. in Width
0.249 to 0.126	0.04T[a]	0.05T[a]	0.05T[a]	0.05T[a]	0.06T[a]
0.125 to 0.092	0.0045	0.005	0.005	0.005	0.007
0.091 to 0.065	0.003	0.004	0.004	0.005	0.006
0.064 to 0.041	0.003	0.004	0.004	0.005	0.006
0.040 to 0.033	0.0025	0.003	0.004	0.004	0.005
0.032 to 0.026	0.0025	0.003	0.004	....	....
0.025 to 0.021	0.0025	0.003	....	....	....
0.020 to 0.019	0.002	0.003	....	....	....
0.018 to 0.015	0.002	....	....	....	....
0.014 to 0.010	0.0015	....	....	....	....

	Over 60 to 66 in. in Width	Over 66 to 72 in. in Width	Over 72 to 84 in. in Width
0.249 to 0.126	0.08T[a]	0.10T[a]	0.11T[a]
0.125 to 0.092	0.010	0.012	....
0.091 to 0.065	0.008	0.010	....

[a] T = the nominal thickness.

Tentative Specifications for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 38 T):[1]

Delete alloy 1 and its specified requirements from Tables I and II and also the information for this alloy given in the table of supplementary data in the Appendix; change the reference to alloy 1 in Section 1 to read "1A."

Tentative Specifications for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 - 40 T):[2]

Table II.—Change the section of this table on permissible variations covering flat sheet from its present form to read as prescribed in the accompanying Table VII.

Tentative Specifications for Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 - 38 T):[1]

Section 1.—Change the third sentence from its present form to read as follows: "Twelve alloys are specified and are designated alloys 1A to 11, inclusive."

Table I.—Delete alloy 1 and its specified requirements from this table.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Specifications for Magnesium Ingot and Stick for Remelting (B 92 - 39):[1]

Section 3.—In the tabulation of chemical properties, change the line reading "total of copper and nickel, max., per cent....0.05" to read as follows:

Copper, max., per cent	0.05
Nickel, max., per cent	0.01

APPENDIX II

A HIGH STRENGTH ALUMINUM SAND CASTING ALLOY NOT REQUIRING SOLUTION TREATMENT AND QUENCH

Prepared for Subcommittee II by D. Basch[1]

Since the use of high-strength aluminum sand casting alloys was instituted, difficulty has been experienced from time to time due to the high-temperature solution treatment and quench necessary to attain the high physical properties. Warping and cracking due to the high stresses introduced by drastic quenching from the neighborhood of 530 C. have not been uncommon, especially where the casting design included long, thin sections or rapid changes in section. Production delays were also frequently encountered due to the following reasons:

1. Actual length of time required for solution and precipitation treatment,
2. Difficulty of fitting solution and precipitation treatment of castings into a foundry production schedule with long waiting lists due to the heavy demands on production capacity of installed high-temperature heat treating equipment, and
3. Scarcity of high-temperature heat treating equipment large enough to handle castings more than about 3 ft. in any direction.

The recent increase in production due to the Defense Program has served to magnify these latter difficulties attendant with the use of the solution-treated, quenched and aged type of high-strength aluminum sand casting alloy. The need for an aluminum sand casting alloy which has physical, chemical and foundry properties comparable with those of the high-strength alloys, but obtainable without the use of a high-temperature solution treatment and quench has been very apparent.

An aluminum-zinc-magnesium alloy has been used as a general purpose sand casting alloy for several years, and recent development work has shown that an alloy with a similar composition satisfied the need stated above. This alloy which is proposed for inclusion as alloy "O" in the A.S.T.M. Tentative Specifications for Aluminum-Base Alloy Sand Castings (B 26 – 37 T) has the following composition:

Zinc, per cent	4.80 to 5.70
Chromium, per cent	0.40 to 0.60
Magnesium, per cent	0.40 to 0.60
Titanium, per cent	0.10 to 0.25
Iron, max., per cent	1.00
Copper, max., per cent	0.20
Silicon, max., per cent	0.60
Other impurities, max., per cent	0.05 each
Aluminum, per cent	balance

The physical properties which have been established as guaranteed specification values for this alloy are as follows:

	As-Cast[a] (unaged)[b]	Artificially Aged (no quench)[c]
Tensile strength, min., psi	29 000	30 000
Yield strength (0.2 per cent offset), psi.:		
min.	..	22 000
typical	16 000	25 000
Elongation in 2 in., min., per cent	6	3

---

[a] The properties are the same as those specified for alloy G, heat treatment No. 1, in A.S.T.M. Specifications B 26-37 T, and are better than alloy M, heat treatment No. 1.

[b] This condition will prevail for two to three weeks after casting. It is only temporary as indicated in the description of the properties of this alloy.

[c] The properties are about the same as those specified for alloy G, heat treatment No. 2, and alloy M, heat treatment No. 2, A.S.T.M. Specifications B 26 – 37 T with somewhat higher yield strength.

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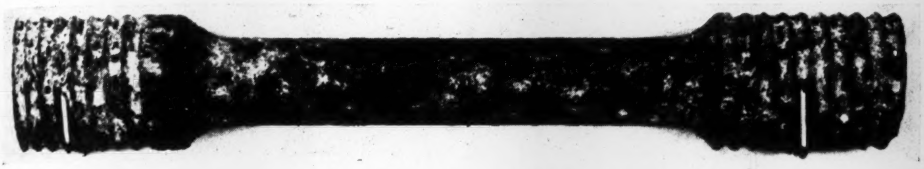
[1] Works Laboratory, General Electric Co., Schenectady, N. Y.

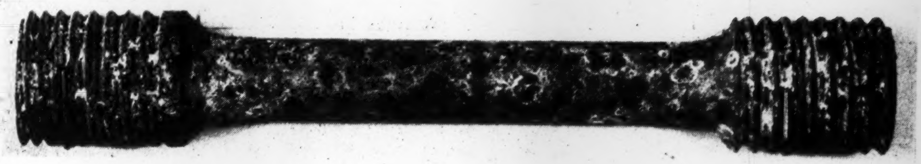
In this condition the proposed alloy meets in tensile strength, elongation and yield strength the requirements prescribed for alloy G, heat treatment No. 2, Specifications B 26 – 37 T, and exceeds those of alloy M, heat treatment No. 2. The high elongation of the proposed alloy in the unaged condition during the first two or three weeks after casting will be useful primarily where cold-deformation, straightening, etc., of castings are necessary. However, even without artificial aging the properties of the alloy will gradually—within three to six months—*approach* the properties of the same casting after artificial aging and lose a great deal of the original ductility just like heat treatment No. 1 when it changes—through air aging—into the aged condition, heat treatment No. 2. The properties of the artificially aged material are those to be counted on in service and, therefore, all design should be based on artificially aged material.

Artificial aging not only stabilizes the physical properties but also serves as a strain relief and permits close tolerance machining within a few days after casting without the danger of subsequent warping or distortion.

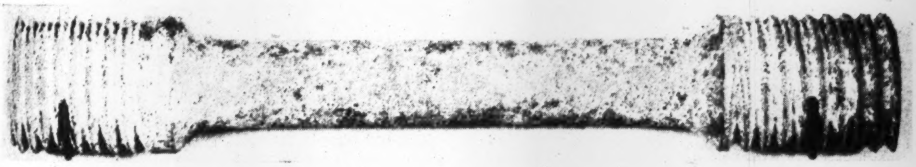
In Table I are shown some typical test data obtained on room temperature



Alloy G, Heat-Treatment No. 2.



Alloy M, Heat-Treatment No. 2.



Alloy O, Artificially Aged.

FIG. 1.—Sand Cast Aluminum Alloy Tension Test Bars After 18 Weeks in Salt Spray (Corrosion Products Removed by Scrubbing).

TABLE I.—TYPICAL TEST DATA FOR ALLOY O.

	Tensile Strength, psi.	Yield Strength (offset 0.2 per cent), psi.	Elongation in 2 in., per cent
AIR AGING ONLY (AVERAGES OF FOUR TESTS)			
10 days..........	30 670	17 770	8.9
60 days..........	32 120	21 520	5.7
120 days..........	36 000	25 750	4.7
AFTER THE ARTIFICIAL AGING TREATMENT			
Individual test bars picked at random	30 900	25 400	4.5
	30 900	25 400	4.45
	30 700	25 700	4.4
	31 000	26 000	4.4
	32 500	24 300	3.9
	32 200	24 100	4.6
	32 500	27 700	3.5
	32 100	27 900	3.2

aged and artificially aged bars of the proposed composition. The stress-strain data were taken with Templin automatic recorders of high sensitivity.

The endurance limit of alloy O in the artificially aged condition has been found to be 8500 psi. Impact resistance as measured by tension-impact tests compares favorably with alloy G, heat treatment No. 2 and is markedly better than alloy M, heat treatment No. 2.

Corrosion tests run by various agencies like the Navy Department, General Electric Co., etc., indicate that the corrosion resistance of the proposed alloy is better than that of alloy G, heat treatment No. 2 of Specifications B 26 – 37 T, although perhaps not quite so good as the silicon-bearing alloys like alloy M, heat treatment No. 2 of Specifications B 26 – 37 T. The corrosion of alloy G, heat treatment No. 2; alloy M, heat treatment No. 2; alloy O, artificially aged; and several others after 18 weeks in the salt spray test are shown in Fig. 1.

Foundry properties are comparable to A.S.T.M. alloy G, but not quite so good as A.S.T.M. alloy M.

X-ray pictures taken on quite complicated castings have demonstrated remarkable soundness of the section at critical points. Machinability is excellent, far superior to either alloy G, heat treatment No. 2, or alloy M, heat treatment No. 2 and probably even better than the standard A.S.T.M. alloy C.

The proposed alloy lends itself very well to anodization with a good white color and high corrosion resistance.

Upward of 225,000 lb. of good castings of this alloy have been made during 1940 with very satisfactory results.

The outstanding advantages of this alloy are the following, which characteristics will indicate its fields of application:

1. High corrosion resistance,
2. Excellent shock resistance,
3. High endurance limit,
4. Excellent machinability, and
5. Procurement of high physical properties comparable to those offered by the so-called heat treatable aluminum alloys now on the market, but without the need of high-temperature solution treatment with its attending quenching stresses which usually result in dimensional instability.

   This omission of the high-temperature solution treatment also eliminates the need for expensive high-temperature heat treating equipment and bottleneck in production.

Against these advantages, there is slightly greater casting difficulty than with the aluminum-silicon alloys due to greater hot shortness and lower strength at high temperatures. These foundry difficulties will require more care in gating and risering than in the case with the high-silicon alloys.

As to the consumers' demands, this is a matter of speculation; however, at a meeting held in New York City some time ago with representatives of the Bell Telephone Laboratories, Sperry Gyroscope, Eclipse Aviation, American Can Co., Ford Instrument, and General Electric Co., it was the consensus of opinion that an alloy with the properties of the proposed composition, which would not require high-temperature solution treatment and quench to develop the properties, fills a very definite need and would find considerable use if once standardized. Since that meeting in New York, the Eastman Kodak Co. and the Parker Appliance Co. have, of their own volition, asked to have their endorsement of and their desire to have this alloy standardized added to those who attended the meeting.

APPENDIX III

THE DIVIDER METHOD IN TENSION TESTING OF MAGNESIUM-BASE ALLOYS

By H. W. Schmidt[1]

One of the properties of materials of interest to engineers is the value of the so-called yield strength. An approximation of this value sometimes used is the stress computed from the load at 0.5 per cent elongation. The "divider method"[2] has been proposed as a means of measuring this 0.5 per cent elongation. It is the purpose of this note to present some data on the divider method as it would be applied to magnesium alloys.

An extruded wrought material (A.S.T.M. Designation: B 107 – 40 T, Alloy 8X)[3] and a heat-treated sand-cast material (A.S.T.M. Designation: B 80 – 40 T, Alloy 4)[4] were used in the tests. All test specimens were prepared in accordance with appropriate A.S.T.M. standards. Several modifications of the "divider method" for measuring the elongation under load were used as follows:

Method A (*Punch-Mark Method*).—Light punch marks on a 2-in. gage length were placed on the test specimen. A divider was set at 2.01 in. Load readings were taken when the operator signalled that the apex of the punch mark coincided with the upper point of the divider leg while the lower leg was firmly held in the bottom gage mark. Simultaneously, and independent of the operator, load readings were taken corresponding to 0.5 per cent elongation of an extensometer.

Method B (*Scratch-Mark Method*).—Scratch marks were placed on the test specimen at a gage length of 2 in. A divider was set at 2.01 in. Load readings were taken when the operator signalled that the upper leg of the divider coincided with the upper scratch mark while the lower leg of the divider was held firmly in the bottom scratch mark. A magnifying glass was used when taking these readings. As previously noted, simultaneous load readings were taken at 0.5 per cent elongation of an extensometer.

Method C (*Pull-Out Method*).—Light prick punch marks at 2-in. gage length were placed on the test specimen. The divider was set at 2.00 in. Load readings were taken when the operator signalled that the apex of the cone of the upper punch mark was visible while the lower leg of the divider was firmly held in the lower punch mark. Simultaneous load readings were taken as before at an elongation of 0.5 per cent of an extensometer.

Only operators skilled in the testing of materials were used in the tests. The data in Table I show the results obtained. In each set of results the first column entitled "Stress at 0.5 per cent

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[1] The Dow Chemical Co., Midland, Mich.

[2] See Standard Definitions of Terms Relating to Methods of Testing (E 6 – 36), 1939 Book of A.S.T.M. Standards, Parts I, II, and III.

[3] Tentative Specifications for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 347.

[4] Tentative Specifications for Magnesium-Base Alloy Sand Castings (B 80 – 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 343.

Elongation" is the stress corresponding to an elongation of 0.5 per cent as measured by an extensometer. The second column is the stress corresponding to the elongation as obtained by one of the divider methods. The third column is the error of the divider method with reference to the extensometer method.

TABLE I.—DIVIDER METHOD IN TENSION TESTING OF MAGNESIUM-BASE ALLOYS.

Investigator	Test Specimen	Method A			Method B			Method C		
		Stress at 0.5 per cent Elongation, psi.	Stress by Punch-Mark Test, psi.	Error, per cent	Stress at 0.5 per cent Elongation, psi.	Stress by Scratch-Mark Test, psi.	Error, per cent	Stress at 0.5 per cent Elongation, psi.	Stress by Pull-Out Test, psi.	Error, per cent
EXTRUDED MAGNESIUM ALLOY (A.S.T.M. DESIGNATION: B 107 - 40 T, ALLOY 8X)[3]										
1	a	30 200	22 500	−25.5	31 200	27 500	−11.9	30 200	16 000	−47.0
	b	29 300	26 100	−10.9	31 400	33 100	+5.4	30 800	18 300	−40.6
	c	30 000	25 400	−15.3	31 300	21 900	−30.0	31 700	23 900	−24.6
2	a	31 200	33 400	+7.1	32 400	32 000	−1.2	30 800	18 400	−40.3
	b	30 200	27 900	−7.6	31 200	30 700	−1.6	30 700	17 800	−42.0
	c	31 200	31 900	+2.2	31 100	31 800	+2.3	32 800	20 700	−36.9
3	a	28 600	25 100	−12.2	31 700	33 300	+5.1	32 300	19 700	−38.8
	b	30 200	30 300	+0.3	32 700	35 000	+7.0	31 800	23 500	−26.1
	c	30 200	30 200	0	30 600	25 900	−15.4	32 800	26 700	−18.6
4	a	30 300	30 500	+0.7	30 600	25 100	−17.9	32 000	23 200	−27.5
	b	30 000	30 600	+2.0	31 800	27 700	−12.9	31 700	30 700	−3.2
	c	30 600	27 900	−8.8				31 100	23 600	−24.1
SAND CAST AND HEAT TREATED MAGNESIUM ALLOY (A.S.T.M. DESIGNATION: B 80 - 40 T, ALLOY 4)[4]										
1	a	14 900	15 000	+0.7	15 200	14 900	−2.0	14 500	11 300	−22.1
	b	15 050	13 850	−8.0	14 700	15 700	+6.8	14 400	10 600	−26.4
	c	14 500	14 500	0				14 500	11 100	−23.5
	d	14 400	14 600	+1.4						
	e	14 800	14 650	−1.0						
2	a	14 700	14 600	−0.7	14 500	14 800	+2.1	14 300	12 500	−12.6
	b	14 300	14 700	+2.8	14 100	15 500	+9.9	14 400	12 400	−13.9
	c	14 500	14 400	−0.7	14 100	14 400	+2.1	14 600	11 200	−23.3
	d	14 800	14 000	−5.4						
	e	14 700	13 800	−6.1						
3	a	14 800	14 700	−0.7	14 400	14 200	−1.4	14 700	12 100	−17.7
	b	14 900	15 000	+0.7	14 100	14 100	0	15 300	12 900	−15.7
	c	14 200	14 200	0	13 900	14 300	+2.9	14 500	13 400	−7.6
	d	14 350	14 500	+1.0						
	e	14 800	14 800	0						
4	a	14 400	16 250	+12.9	15 100	13 700	−9.3	15 000	10 700	−28.7
	b	14 550	16 900	+16.2	14 900	14 900	0	14 700	11 300	−23.1
	c	14 000	15 000	+7.1	15 000	15 000	0	14 200	12 100	−14.8
	d	14 250	11 500	−19.3						
5	a	14 300	12 250	−14.3						
	b	14 150	11 700	−17.3						
	c	14 300	16 600	+16.1						
	d	14 250	15 000	+5.3						
	e	14 400	12 850	−10.8						

Discussion:

The results show that while one operator may obtain consistently good results with the punch-mark method A, other operators may obtain very poor results. There is a considerable discrepancy in the results obtained by an operator when testing a cast material as against the same operator testing a wrought material. In using the punch-mark method, it is essential that the prick-punch marks be very light. Good illumination is also a requisite. The sense of feel can be developed as a valuable aid to vision.

The scratch-mark method B gave more consistent results for the cast materials than for the wrought material. This may be explained on the basis that the machined surface of the specimen of wrought material caused confusion since it was difficult to distinguish between the scratch mark and machine marks. This difficulty could be eliminated by polishing the bars longitudinally to remove transverse machine marks. The scratch-mark method lends itself to a greater degree of accuracy than does the punch-mark method. However, for the method to be effective, transverse scratch marks must be removed as these cause confusion and error. With this method, proper illumination is especially essential. Very light scratch marks are necessary.

The pull-out method C is definitely unreliable. The depth of punch marks, size of divider points, size and type of divider used, illumination, and skill of the operator are all variables influencing the accuracy of the method.

The divider methods "A" and "B" may possibly be used with some success by a skilled operator who is thoroughly familiar with the material to be tested and adequately trained in using the method. The divider setting and the manipulation of the divider are important functions of the test and if proper care is not exercised, large errors may result. The methods are not recommended.

Conclusions:

The divider method is not recommended for use as a control method in tension testing of magnesium alloys.

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS

The complications in procedure in the preparation of specifications for electrodeposited metallic coatings developing out of the number of standing committees interested in the subject, became sufficiently serious during 1940 to require attention. Committees A-5 on Corrosion of Iron and Steel, B-3 on Corrosion of Non-Ferrous Metals and Alloys, and B-6 on Die-Cast Metals and Alloys relinquished that portion of their scope covering electrodeposited coatings, with the exception in the case of Committee A-5 of zinc and cadmium coatings on steel, in order to make possible the formation of a new standing committee on electrodeposited metallic coatings. The Executive Committee of the Society felt this move to be desirable and a steering committee composed of W. H. Finkeldey, Sam Tour, J. R. Townsend, William Blum, C. H. Sample, and E. A. Anderson was appointed to undertake the organization of the committee. Mr. E. A. Anderson was requested to act as temporary chairman. His effective work made possible the rapid organization of and action by the committee.

On March 6, 1941, Committee B-8 on Electrodeposited Metallic Coatings was formally organized with the following elected officers:

Chairman, E. M. Baker
Vice-Chairman, E. A. Anderson
Secretary, G. Soderberg

The membership of the committee totals 31, of whom 12 are classed as producers, 4 as consumers, and 15 as general interest members.

The existing Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 40 T) and the Tentative Methods of Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 – 40 T) were released from the jurisdiction of Committee A-5, and the responsibility for them was assumed by Committee B-8.

I. New Tentative Standards

The committee recommends that the following two new, proposed specifications, which had previously received preliminary discussion by Subcommittee IV on Specifications, of Committee B-3, and Subcommittee VI on Finishing Properties of Die Castings, of Committee B-6, be accepted for publication as tentative:

Tentative Specifications for:[1]

Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys, and

Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys.

II. Revisions of Tentative Standards

Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 40 T).[2]—The committee recommends that these specifications be revised as appended hereto[3] and continued as tentative.

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[1] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, pp. 559 and 562.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 296.

III. EDITORIAL CHANGES IN TENTATIVE STANDARD

Tentative Methods of Test for Local Thickness of Electrodeposited Coatings on Steel (*A 219 - 40 T*).[4]—The committee recommends the following changes in these methods as editorial:

Title and Scope.—Omit the words "on steel."

Section 5.—Correct the concentration of the hydrochloric acid specified from a sp. gr. of "1.80" to read "1.180."

Section 6 (*a*).—In the second sentence change the phrase "small ring of melted paraffin or with a mark made with a wax pencil," to read "small ring made with melted paraffin or with a wax pencil."

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 30 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-Base Alloys.........	28	0	2
Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys................	26	0	4
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 40 T)....................	28	0	2
III. EDITORIAL CHANGES IN TENTATIVE STANDARD			
Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 - 40 T)......	27	0	2

This report has been submitted to letter ballot of the committee which consists of 30 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. M. BAKER,
Chairman.

G. SODERBERG,
Secretary.

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[3] These revised specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 555.

[4] 1940 Supplement to Book of A.S.T.M. Standards Part I, p. 303.

REPORT OF COMMITTEE C-1

ON

CEMENT

Committee C-1 on Cement has held two meetings during the year. Each of these occupied two entire days during which all of the committee's various activities were very fully discussed. Most attention, however, was given to questions which largely concerned the Tentative Specifications for Portland Cement (C 150 - 40 T) covering requirements for five types of portland cement. As a result of this consideration, the committee now recommends that Specifications C 150 be revised as appended hereto[1] and approved for reference to letter ballot of the Society for adoption as standard to replace immediately the Standard Specifications for Portland Cement (C 9 - 38)[2] and for High-Early-Strength Portland Cement (C 74 - 39).[3] This revision was prepared after detailed study of the specifications by the Sponsoring Committee on Portland Cement under the chairmanship of C. E. Wuerpel.

Though it will be noted that apparently but slight revisions have been made in Specifications C 150 - 40 T, they have been published as tentative for a year, and it should be stated that much thought was given to a number of proposed revisions, such as changes in strength requirements, in chemical limitations, fineness, definition, statements regarding permissible additions, and the insertion of maximum limits on the permissible soda (Na_2O) and potassa (K_2O) contents.

SUBCOMMITTEE ACTIVITIES

Alkali (Na_2O, K_2O) in Cement:

Attention has been directed in various articles during the past few years to the possibility of the reaction of "alkali," set free in the hydration of cement, on certain minerals occurring in aggregate. In several cases in highway work in California and in the Parker Dam on the Colorado River the resulting action manifested itself either by "map cracking" on the surface, or by marked cracking extending to some depth in the concrete, or marked volume changes amounting to incipient disintegration. Although the alkali present to a major extent in the hydration of cement is lime (CaO), which doubtless does react with all but calcareous aggregate—and in so doing may not produce unfavorable results—the evidence advanced to date seems to place the fault with the Na_2O and possibly also the K_2O which is present in all cements. Although the total amounts of these oxides never exceed 1.5 per cent—and may be as low as 0.10 per cent—and averages possibly for all cements approximately 0.75 per cent, it appears that amounts of about 0.60 per cent are sufficient to cause concern, particularly with rocks or minerals such as andesite, opaline-like silicas, possibly certain cherts and feldspars, indurated shale-like rocks, etc.

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[1] These specifications were adopted as standard by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 1.

[3] *Ibid.*, p. 3.

As a result of the agitation of this question, the committee last year considered the need of limits for the Na_2O and K_2O before proposing the Tentative Specifications C 150 - 40 T. However, it was believed the question should be studied further. There was organized during the year a subcommittee to look into the matter, under the direction of R. F. Blanks. At the time of the last meeting of Committee C-1, this subcommittee was not able to advance any recommendations, and hence in the revision of Specifications C 150, submitted herewith[1] for adoption as standard, there are no suggested limits for the two oxides in question. However, all of the members of the committee are mindful of the fact that this is one of the most important technical matters now before the cement industry and the subject will have the committee's marked attention.

Chemical Limitations and Analyses:

The Subcommittee on Methods of Chemical Analysis under the chairmanship of W. C. Hanna, has been, as usual, very busy. Though at this time it has no revisions to suggest, it is actively studying the method of determining free lime, sulfide sulfur, Na_2O, and K_2O. During the coming year it will devote much attention to better and quicker methods for determining the two last mentioned oxides. This subject will be discussed in some detail later under the activities of the Cement Reference Laboratory.

The following editorial changes in the Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 40 T) are recommended for inclusion in the methods when they are next printed:

Section 1 (b).—Change the last four sentences from their present form to read as follows:

> The solvent should be slightly alkaline (Note 3) to the indicator. If the solvent is colorless, add a diluted solution of NaOH in absolute ethanol until a slight pink color appears. If the initial color is pink, remove it with the standard solution of ammonium acetate and add the solution of NaOH until a slight pink color appears. Heat 60 ml. of the solvent to boiling. If the pink color persists, add one small drop of the standard solution of ammonium acetate (0.02 ml., equivalent to 0.0001 g. CaO) to the hot solvent. If the pink color does not disappear, the solvent contains too much alkali, which should be reduced. The solvent, if allowed to stand for a considerable length of time, may become slightly acid and should be checked and, if necessary, readjusted from time to time. (Note 4.)

Add the following note as a new Note 3, renumbering the remaining notes accordingly:

> NOTE 3.—While a solvent, which is exactly neutral at the boiling point, is the ideal, it is difficult to prepare due to the end point not being sharp and the danger of adding too much ammonium acetate. The error due to slight alkalinity is not more than 0.01 per cent in a determination of free calcium oxide and may be disregarded. In making up the solvent, the end point may be better observed if the solvent is put in a tall cylinder placed on white paper. The solvent may be observed from the top and the color matched with that of acidified solvent in a similar cylinder.

Section 1 (c).—Omit the phrase "for 20 min." from the fifth and eighth sentence, and the phrase "20 min." from the ninth sentence.

Add a reference to a new Note 8, to read as follows, at the end of the ninth sentence and renumber the present Note 7 as Note 9:

> NOTE 8.—In general the intervals may be 20 min. but they will chiefly depend on the rapidity of the dissolution of calcium oxide. They may be 5 to 10 min. in the beginning and then be increased to 30 min. or 1 hr. in the end. If the solution contains a large amount of dissolved calcium oxide and its boiling is continued a long time without titration, crystals (probably calcium glyceride) may form which dissolve slowly and increase the time required for the completion of the titration.

Note 6.—After the fourth sentence, add a new sentence to read as follows:

> Instead of an ordinary flask, plain tubing, and a stopper, a flask with a ground-in glass stopper and condenser tube may be used, preferably one with a standard taper joint.

Note 7.—At the end of the first sentence add the phrase "especially when a large amount of calcium acetate is present."

Section 2.—In Note 1, second paragraph, change the last two sentences from their present form to read as follows:

> Mix all the screenings and ground residue thoroughly, grind slightly over 1 g. rapidly in the agate mortar for 2 or 3 min., and take 1 g. for the determination of free calcium oxide. Avoid unnecessary exposure of the sample to the atmosphere. If the sample is fine enough to pass completely through the sieve, grind slightly over 1 g. as above before weighing 1 g. for the determination.

In Note 2, add the following two sentences between the first and second sentence:

> To avoid such excess, use a small burette or measuring pipette fitted with a glass stopcock or a rubber pinchcock and small tip which delivers about 50 drops per milliliter. If the titrations are far apart and the reagent evaporates in the tip, a few drops may be discarded before each titration and the drops used in titration may be counted and converted in terms of milliliters.

Add a new paragraph to read as follows between the first and second paragraphs in Note 2:

> Vigorous boiling is more essential with cement than with pure calcium oxide and, if possible, should be so conducted that it is not necessary to shake the flask vigorously at intervals. The flask should not be shaken vigorously while it is disconnected from the condenser because the solvent, if superheated, may boil, expel alcohol vapor, and then draw in air carrying water and carbon dioxide. A good procedure is to bring the solution and sample to boiling rapidly over a bunsen flame in the beginning before placing the flask on a hot plate and connecting it with the condenser. If there is a tendency to bump, it may be reduced by adding glass beads or pure quartz sand to the flask or putting a thin mat of asbestos between the flask and the hot plate.

In Note 2, second paragraph, delete the fourth sentence and add at the end of the paragraph a new sentence to read as follows:

> The contents of the flask may be matched with similar contents in another flask which contains an excess of ammonium acetate.

Section 3 (b).—Add a reference to a new note after the third sentence and add the following note at the end of the paragraph:

> NOTE.—A convenient way to prepare a large amount of solvent is to heat $BaCl_2 \cdot 2H_2O$ at 120 to 130 C. for several hours, dissolve it in hot glycerol (100 to 125 C.) without grinding, and mix the solution with ethanol. If the salt is dehydrated at a much higher temperature, it may be slow to dissolve in hot glycerol or solvent even when ground.

Section 4 (b).—Delete this paragraph, relettering the remaining paragraph accordingly.

Volume Change and Soundness:

The Subcommittee on Volume Change and Soundness, under the chairmanship of H. F. Gonnerman, has been studying those questions under its jurisdiction, and has been following work which is being done along those lines by the laboratories of the Portland Cement Association and the Public Roads Administration. The latter has been actively studying the autoclaving of clinker as proposed by Ira Paul of the New York State Department of Public Works. The clinker of 60 cement mills has been subjected to autoclaving, the results compared with the expansion of neat cement bars, freezing-and-thawing tests, the Merriman sugar test, the usual acceptance tests of cement, etc. No conclusions have yet been developed, but some valuable information should accrue during the coming year. It should be noted that in the revision of Specifications C 150 the old pat test for soundness, which has been in existence since the first standard for portland cement was adopted by the Society, has been supplanted by an autoclave test. The Federal specifications for portland cement are being similarly revised.

Sulfate Resistance:

The Subcommittee on Sulfate Resistance, under the chairmanship of Dalton G. Miller, has a number of interesting problems before it. One of the types of cement in Specifications C 150, as recommended for adoption at this time, is referred to as a sulfate-resisting cement. The committee, however, feels considerable concern regarding the adequacy of the specification with regard to this particular cement. At the present time the outstanding requirements which are used in the specification as an indication of sulfate resistance are indicated solely by chemical limitations without the use of any physical tests. There is a questioning on the part of many as to the correctness of these chemical limitations. In view of the desire for a physical test indicative of sulfate resistance and the correlation of such a test with chemical requirements, the subcommittee has outlined a series of tests. To study the relative volume change of lean mortar bars when subjected to the attack of aggressive sulfate solutions, approximately 125 different cements will be included in the investigation.

Strength:

As a result of data submitted by the National Bureau of Standards, the Subcommittee on Strength (O. L. Moore, chairman) is studying the possibilities of the vibration method for placing mortar in the molds for compressive strength specimens. Tests are already in progress in five laboratories. Each of the five types of cement described in Specifications C 150 will be used with two vibrators which have been tried in a preliminary way. One vibrator is of the electromagnetic type; the other the rotary type. One of the vibrators is commercially available while the other follows a design used by the National Bureau of Standards. In the Bureau tests a speed of 5000 rpm. had been found preferable for the rotary vibrator, but in the work now being sponsored by Mr. Moore's subcommittee a speed of 3450 rpm. is being used for the rotary vibrator since most laboratories have alternating current. If a greater speed is found desirable, the rotary vibrator will have to be redesigned. The purpose of molding by vibration is to remove variables induced by manual molding. It is of interest to note that the latest British specifications for portland cement specify making compression tests by vibration.

Because the above described investigation of placing mortars by vibration may lead to revisions of the method, Committee C-1 recommends that the Tentative Method of Test for Compressive Strength of Portland-Cement Mortars (C 109 – 37 T) be retained as tentative for another year.

The committee has also given thought to the possibility of the machine mixing of mortars for test specimens. The Subcommittee on Strength completed an assignment to collect certain strength data which were desired by the Sponsoring Committee on Portland Cement.

Consistency:

The Subcommittee on Consistency (M. A. Swayze, chairman) has been studying the possibility of determining the amount of water to be employed in making mortar by testing a mortar rather than the neat paste used at present. While the results look interesting, further work is needed before recommendations can be made. The subcommittee has also been stressing the need of more care in mixing so as to reduce the loss of water and water-cement paste during the period in which the manipulation is carried out. The need of mechanical equipment is recognized and will be considered. In this connection W. H. Klein exhibited a rather small rubber bag which he has been trying out in the mixing of mortars. The ingredients for mortar are placed in the bag and then kneaded manually or mechanically. Such a closed and relatively flexible container warrants considerable further trials.

Fineness:

The Subcommittee on Fineness (P. H. Bates, chairman) is recommending certain revisions in the Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 - 38 T). It does not recommend the adoption of this method as standard at the present time. The subject of the fineness of cement and the need of such a requirement in standards is still a moot question. This matter received considerable thought in the committee's consideration in recommending the adoption of revised Specifications C 150 as standard. Many ideas were presented, but the consensus of opinion was for retaining the fineness limits as given in the recommended standard. The committee is, therefore, recommending the adoption of the above mentioned revisions of the Tentative Method C 115 - 38 T which have been incorporated in the revised method appended hereto.[4] The revisions do not cover any marked changes in the procedure followed in using the method. They are mostly in the nature of rewording suggested for the sake of clearness.

During the year there appeared in the ASTM BULLETIN[5] an article dealing with the determination of the fineness of cement and other finely divided materials by means of air permeability measurements. This matter was further discussed at the Symposium on New Methods for Particle Size Determinations in the Subsieve Range held at the Washington spring meeting of the Society on March 4, 1941. During the year the Subcommittee on Fineness received the cooperation of ten laboratories in the use of this method for determining the fineness of five portland cements, one portland-pozzuolana cement, and one cement raw mixture. The results are given in Tables I and II. Considering the lack of familiarity with the method, the precision of the results indicates the desirability of continued work. This will be done.

Additions and Tests of Additions to Cement:

During the year the Subcommittee on Tests of Additions to Cement (H. J. Gilkey, chairman) completed its study of the addition of TDA to portland cements other than the high-early-strength type. As a result, the committee has placed in a note to Section 3 of the revision[3] of Specifications C 150 a statement to the effect that this addition is not considered harmful in the amounts given.

Another producer of an addition has requested that the Society study the product which he is making when used in cements for highway paving concrete. This study is being undertaken, particularly since that addition is being used in cement in a considerable amount in highway construction by the State of New York and is being made the subject of investigation by the highway departments of several other states. The committee is also following the results being obtained by some cement producers and by consumers of cement when using cements containing such fats as tallow and certain stearates. The very excellent resistance to frost action, especially in the presence of calcium chloride, which is being noted in the highways of New York where certain additions have been used compels the committee to give special attention to such additions as seem to incorporate a small percentage of air into the concrete.

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[4] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 257.

[5] R. L. Blaine, "Studies of the Measurement of Specific Surface by Air Permeability," ASTM BULLETIN, No. 108, January, 1941, p. 17.

TABLE I.—SPECIFIC SURFACE OF CEMENTS BY AIR-PERMEABILITY METHOD AS REPORTED BY VARIOUS LABORATORIES, SQ. CM. PER G.

Laboratory	Cement 1	Cement 2	Cement 3	Cement 4	Cement 5	Cement 6[b]	Cement 7[c]
No. 1	3215	3565	3675	2500	5140	5170	5340
No. 2	3245	3460	3600	2504	5230	4996	5628[d]
No. 3	3182	3488	3542[a]	2474	5100	5088	5306
No. 4A	3090	3410	3530	2430	5020	4960	5180
No. 4B	3180	3500	3620	2470	5110	5070	5330
No. 5	3170	3510	3630	2540	5160	5090	5250
No. 6	3230	3540	3670	2545	5200	4980	5100
No. 7	3063	3404	3482	2426	5008	4903	5059
No. 8	3194	3491	3617	2489	5166	5113	5290
No. 9	3095	3425	3509	2426	4987	4914	5078
No. 10	3120	3380	3550	2460	5080	5060	5210
Average	3162	3470	3584	2478	5109	5031	5252
Percentage variation	1.64	1.30	1.56	1.37	1.25	1.45	2.17

BY USING CEMENT NO. 1 AS A STANDARD SAMPLE WITH A FINENESS VALUE OF 3200 SQ. CM. PER G. AND APPLYING FACTOR TO VALUES REPORTED BY EACH LABORATORY

Laboratory	Cement 1	Cement 2	Cement 3	Cement 4	Cement 5	Cement 6[b]	Cement 7[c]
Average	3200	3510	3630	2510	5170	5090	5310
Percentage variation	....	0.83	0.72	0.80	0.68	1.22	1.33

DIFFERENCE BETWEEN HIGHEST AND LOWEST VALUES AS REPORTED BY LABORATORIES, SQ. CM. PER G.

Laboratory	Cement 1	Cement 2	Cement 3	Cement 4	Cement 5	Cement 6[b]	Cement 7[c]
No. 1	20	10	50	10	15	50	55
No. 2	136	174	164	195	136	478	532
No. 3	20	60	400[e]	70	70	70	40
No. 4A	50	40	10	10	10	50	90
No. 4B	0	0	10	10	20	30	20
No. 5	60	30	50	40	40	40	40
No. 6	11	18	26	41	62	270	126
No. 7	66	90	52	24	26	93	164
No. 8	5	27	43	34	109	109	245
No. 9	15	50	46	15	47	35	104
No. 10	40	100	70	70	60	80	20

[a] One bad value, average of the other four determinations was 3620 sq. cm. per g.

[b] Portland-pozzuolana (calcined clay).

[c] Raw mix (Lehigh Valley).

[d] Using 13-g. sample, value of 5263 sq. cm. per g. was obtained.

[e] One bad value, spread of the other four determinations was 20 sq. cm. per g.

Portland-Pozzuolana Cements, Masonry Cements:

The subcommittees having jurisdiction over such cements have spent considerable time going over the Society's present specifications for these commodities. The nature of both of these types of cement varies over quite a range, and consequently standard requirements and methods of tests tend to become rather involved if they adequately cover the entire range. This makes the preparation of standards rather exacting and laborious. However, the increased use of both of these materials, especially, masonry cement, necessitates the attention which the subcommittee is giving to this work.

TABLE II.—SPECIFIC SURFACE OF CEMENTS AS DETERMINED BY THE WAGNER AND KLEIN TURBIDIMETERS AND ANILINE HYDROMETER, SQ. CM. PER G.

Method Used	Cement 1	Cement 2	Cement 3	Cement 4	Cement 5	Cement 6	Cement 7
Wagner Turbidimeter	1729	1938	1873	1382	2612	2026	2197
	1650	1840	1830	1350	2590	1840	2840
	1628	1918	1770	1263	2510	1868	3065 2675
	1690	1890	1820	1390	2500	1930	2610
	1652	1896	1815	1265	2542	....	....
Klein Turbidimeter	1460	1620	1450	1340	2180	2120	....
Aniline Hydrometer	1972	2159	2002	1602	2743	2220	....

Miscellaneous Committee Activities:

The several subcommittees on bleeding, heat of hydration, time of setting, and new tests, have been more or less actively engaged in their work. While they do not have results at hand of sufficient interest to justify publication, they all have been closely following developments of interest to their work.

Cement Reference Laboratory:

The Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman) has submitted the following summary of its work for the period July 1, 1940, to February 28, 1941:

The sixth inspection tour among cement testing laboratories was completed and a seventh tour was announced and begun. Requests for inspection were received from 267 laboratories. The number of laboratories which had requested inspection in the sixth tour was 290.

Many of the requests for inspection have mentioned improvements made in apparatus and methods since the last inspection. Some of the deficiencies remedied were of less importance than others, but the general indication was that of a closer regard for specification requirements. Efforts were made to avoid unwarranted inspections, and some few laboratories, which had not corrected major deficiencies mentioned in the reports of earlier inspections, were advised that their requests for reinspection were only tentatively listed pending developments.

The Public Roads Administration continued to require Cement Reference Laboratory inspections at laboratories involved in the testing of cement for certain Government projects. Copies of inspection reports have also been furnished to other interested supervising offices.

Two comparative test samples of portland cement, Nos. 4 and 5, were distributed to 225 laboratories for the purpose of comparing the results of chemical analyses. The test data from 188 laboratories were summarized by the Reference Laboratory into a report which was issued by the National Bureau of Standards to the participating laboratories. In some cases the results were not received until much later than anticipated, probably largely on account of the unusual demands upon and changes in the staffs of some of the laboratories during recent months. The work included determinations of SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, ignition loss, Na_2O, K_2O, P_2O_5, Mn_2O_3, insoluble residue, free CaO, and water-soluble alkali.

The report on comparative samples Nos. 4 and 5 also included brief summaries of the data on balances and weights used in the work. In recent years there has been occasional discussion about such equipment, and it is hoped that the data now presented will be helpful in future considerations of the analytical balances and weights used in the cement laboratories.

The examination of molds used for 2-in. cubes was made a part of the regular work. Also, with the start of the seventh tour, the verification of compression testing machines was begun.

The laboratory completed the last of the required 1-yr. strength tests on specimens made with a cement containing a certain addition, added during grinding of the cement, and submitted the results to the Subcommittee on Tests of Additions to Cement.

RECOMMENDATIONS AFFECTING STANDARDS

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 83 voting members; 73 members returned their ballots, with the results shown in Table III.

TABLE III.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 – 38 T): Revisions mostly for clarification; not making any marked changes in method.	68	0	4
II. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Spec. for Portland Cement (C 150 – 40 T) as revised, to replace Standard Specifications for Portland Cement (C 9 – 38) and for High-Early-Strength Portland Cement (C 74 – 39)..........	63	4	6

This report has been submitted to letter ballot of the committee, which consists of 83 voting members; 73 members returned their ballots, of whom 70 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

P. H. BATES,
Chairman.

L. W. WALTER,
Secretary.

DISCUSSION

Mr. Duff A. Abrams[1] (*by letter*).—The writer takes exception to the paragraph, of the report of Committee C-1, under the heading "Subcommittee Activities" dealing with the effect of alkalies in cement on the durability of concrete. He attended both meetings of Committee C-1 during the year covered by this report and received all data that were presented to the committee, but saw nothing in the way of subcommittee activities which would justify the published statement.

It is unfortunate that Committee C-1 did not identify the "various articles during the past few years" on which its conclusions were based and give an estimate of the sufficiency of the information contained therein. The earliest recent article on this subject that has come to my attention was written by T. E. Stanton in *Engineering News-Record*, February 1, 1940. Assuming that the report of the committee was completed about May 1, 1941, "the past few years" covered exactly 15 months. The writer is aware that statements have been quoted (also mis-quoted) from *Bulletin 182* of the U. S. Geological Survey (1901) and from a paper by A. E. Stephenson in the *Journal of Geology*, February–March, 1916, in an attempt to furnish a pedigree for certain recent hypotheses. However these papers have no recognizable bearing on the case of portland cement concrete. Is it possible that the committee's "past few years" might include articles 40 and 25 years old?

One cannot avoid noting the extreme caution of certain statements of the committee on the subject of alkali in cement. In line 3, there was "the possibility of the reaction of alkali"; however by the time we reach line 8, "the resulting reaction manifested itself." Caution again returns so that in line 20 it "seems to place the fault with the Na_2O and possibly with the K_2O which is present in all cements"; in line 26 "it appears that amounts of about 0.60 per cent are sufficient to cause concern."

It is notable that the committee had no comments on the following features of this subject:

1. All cements contain the so-called alkalies; it is doubtful if a commercial portland cement was ever produced without them; hence alkalies in portland cement concrete is no novelty.

2. It has been known for nearly a century that alkalies were present in portland cements. The earliest chemical analysis of a portland cement which has come to my attention was made in 1849 on an English cement by a German chemist; it was reported[2] as follows:

Ca	54.1
Si	22.2
Al	7.7
K	1.1
Na	1.6
Fe	5.3
C_2	2.1
Aq	1.0
	95.1

While the analysis is somewhat incomplete it represents a most creditable performance for 1849; it is probably as accurate as are many modern analyses which, without having determined alkalies or other minor compounds report total oxides varying from 99.9 to 100.3 per cent for cements which are known to contain $\frac{1}{2}$ to $1\frac{1}{2}$ per cent of alkalies.

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[1] Consulting Engineer, New York, N. Y.

[2] A. Lipowitz, "Practical Manufacture of Portland Cement," p. 13, sample 3. Book written 1867, translated from German by W. F. Reid, and bound with Reid's book on the same subject by Baird, Philadelphia (1869). Gives other early chemical analyses of portland cements.

K and Na in the above analysis are interpreted to be the compounds now designated as K_2O and Na_2O. On this basis the combined alkalies is 2.7 per cent. The computed compound composition is: C3S = 0 per cent, C2S = 64 per cent, C3A = 11 per cent. The presence of water (Aq) and C_2 (probably CO_2) suggest a considerable percentage of raw mix in the finished cement or that the sample had been stored in a damp place.

3. The alkalies are rarely mentioned in cement specifications; most chemical analyses have disregarded them. It is only recently that the A.S.T.M. suggested a method for their determination.

4. In the hundreds of thousands of tests made on portland cements in the numerous elaborate experimental studies of special cements carried out about 1930–1933, prior to the construction of Boulder Dam, no consideration was given to alkalies, nothing was learned as to their behavior, and no thought was given to limiting their amount. One of the brands of cement used in Boulder and Parker Dams contained 1.42 per cent alkalies; among the highest in the U. S.

5. The committee failed to comment on the peculiar fact that in recommending special low-heat cements (in which high alkalies is now said to be troublesome) for Boulder, Parker and other large concrete dams, the clock of portland cement progress was turned back nearly a century. The compound composition of the 1849 English cement is not materially different from some of the low-heat cements which have been manufactured in large volumes especially for the huge concrete dams built during the past 8 yr. Some of the cements used in Parker Dam showed C3S as low as 10 per cent; C2S as high as 60 per cent and C3A as high as 7 per cent.[3]

6. During the investigations which were carried out in 1940 by the U. S. Bureau of Reclamation on materials used in Parker Dam, 2 years after the dam was completed, no determination of alkalies in cement could be found and none could be made until samples of the four low-heat cements manufactured in Southern California were finally located in Chicago.

7. In making the chemical analyses for the acceptance tests of about 15,000,000 barrels of cements for Boulder, Grand Coulee, Bartlett and Parker Dams during the years 1933 to 1940 (to mention only a few of the larger concrete dams) the National Bureau of Standards did not report a single alkali determination.

8. The committee failed to note that, allowing for the normal variations in samples and necessary tolerances in the determination of alkalies, about 75 per cent of all American portland cements would be rejected by a limit of 0.60 per cent on combined alkalies.

9. The committee did not comment on the failure of previous geological or petrographic studies of concrete aggregates and foundation rocks to reveal any danger signals for the guidance of builders of large concrete dams.

The first time the question of the effect of alkalies in cement on concrete durability was brought before Committee C-1 was at the Washington meeting of December 6, 1940, when Mr. R. F. Blanks of the U. S. Bureau of Reclamation presented an oral statement concerning the deterioration of Parker Dam which became notable within 2 years after the completion of that $9,000,000 structure.

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[3] From "Summary Report of Bureau of Standards Acceptance Tests for Low-Heat Cement for Bartlett and Parker Dams."

The only material that has thus far come before Committee C-1 in the way of subcommittee action was a "Report of the Working Committee, Effect of Alkalies in Cement on the Durability of Concrete" which was sent to the members of Committee C-1 by Mr. R. F. Blanks, Chairman of the Working Committee, under date of March 11, 1941. Apparently the report had not previously been seen by the members of the Working Committee and contained statements which the members did not consider justified and the report was withdrawn.

The report of Committee C-1 contains statements and conclusions, issued in the name of the committee, on a highly controversial subject, to which I object for the following reasons:

(*a*) No information on recommendations were presented to the committee by the subcommittee dealing with this subject;

(*b*) The sentiments expressed in the report of the committee closely parallel those of the report of the chairman of the Working Committee, which was repudiated by the Working Committee.

(*c*) No scientific data have been presented to Committee C-1 which substantiate any of the important statements or conclusions presented in their 1941 report;

(*d*) Much material was available on this subject which was not brought to the attention of Committee C-1.

(*e*) The committee had no opportunity to reach any conclusions or to take any action on the effects of alkalies in cement on the durability of concrete;

(*f*) I have made a critical study of the "incipient disintegration" of "Parker Dam on the Colorado River" (completed, May, 1938). In this study I examined detailed reports on cements and aggregates, design and construction records, crack and condition surveys and reported studies of the behavior of concrete taken from the dam. None of this information was ever made available to Committee C-1. My conclusions with reference to Parker Dam are entirely different from those stated in the report of Committee C-1 on Cement.

(*g*) The report of the Committee mentions with apparent approval a limitation of 0.60 per cent on alkalies, with little or no information on which to base such a limit, and no hint as to how the manufacturers are to meet such a limit.

(*h*) Committee C-1 is one of the oldest of A.S.T.M. Committees; it was organized in 1902 and has issued reports practically every year since. In only three of these reports is to be found any data on alkalies in portland cement; then only the quantity of alkalies was determined and in every instance the determination of alkalies was only incidental to other objectives. In no annual report of this committee, prior to 1941, is there to be found a single reference to the effect of alkalies in cement on concrete durability.

(*i*) The off-hand pronouncements of Committee C-1 with reference to alkalies in portland cement should be contrasted with the somewhat parallel case of magnesia. The controversy over the effect of magnesia in cement on the durability of concrete has been under way for nearly a century; dozens of papers have been written on this subject, based on thousands of tests, yet the question has never been settled to the satisfaction of anyone and the proper specification limit for MgO is still controversial.

In my opinion the entire statement of the 1941 Report of Committee C-1 on the effect of alkalies in cement on concrete durability is unwarranted and misleading.

REPORT OF COMMITTEE C-5

ON

FIRE TESTS OF MATERIALS AND CONSTRUCTION

A meeting of Committee C-5 on Fire Tests of Materials and Construction was held on March 5, 1941, in Washington, D. C.

I. New Tentative Standard

For many years there has been need for a standard method for testing wood chemically treated by impregnation to determine its fire-retardant properties. Since 1899 the City of New York has by ordinance required wood entering into the construction of buildings exceeding a certain height, in place of prescribed incombustible interior finish, to be "treated by some process (approved by the building authorities) to render the same fireproof." The extensive use of wood, in later years, in important projects, such as the New York World's Fair, and in other fields, has decidedly increased that need. The City of New York, after trying several tests that had been in use by the U. S. Navy Department, adopted a standard test developed by the former chairman of Committee C-5, the late Ira H. Woolson. This standard, generally known as the "timber test," together with the earlier so-called "crib" and "shavings" tests, became one of the criteria of acceptance of treated materials. This test, like the other two, had the advantage of requiring simple but rugged apparatus suitable for prompt determinations and has been in use for many years. Experience, however, has shown that the results have not been entirely consistent and as early as 1927 a study was undertaken by a subcommittee of Committee C-5 to develop a more satisfactory test. The method proposed as a result of this work, referred to as the "fire tube test," gives well-defined distinction between treated and untreated wood and degrees of treatment, but requires specimens not readily obtainable from material delivered for use. After several years of experimentation and discussion, no single method proved acceptable to a sufficiently large part of Committee C-5 to justify adoption. Hence, a study of all current testing methods including a modified "crib test" was undertaken in 1937. Five cooperating laboratories, working independently, conducted an elaborate series of tests of treated and untreated wood the results of which are appended hereto in a Report on Comparative Fire Tests of Treated and Untreated Wood prepared by Subcommittee II on Fire Tests of Lumber (W. J. Krefeld, chairman). Based on this investigation, Subcommittee II has prepared a proposed Tentative Method of Test for Fire-Retardant Properties of Wood as appended hereto.[1] On the recommendation of the subcommittee, Committee C-5 recommends, as a result of action taken at the meeting on March 5, 1941, that the method be accepted for publication as tentative.

II. Adoption of Tentative Standard as Standard

At the annual meeting in June, 1940, Committee C-5 submitted for publication the Tentative Methods of Fire Tests of Door Assemblies (C 152 - 40 T).[2] Subcommittee III on Fire Tests of Wall Opening Protectives (A. L. Brown, chairman) has given further consideration to the tentative methods. No criticisms have been received. On the recommendation of the subcommittee, Committee C-5 recommends, as a result of action taken at the meeting on March 5, 1941, that Tentative Methods C 152 - 40 T be approved for reference to letter ballot of the Society for adoption as standard.

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[1] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 281.

III. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

In the 1940 report the committee presented a tentative revision[3] of the Standard Specifications for Fire Tests of Building Construction and Materials (C 19 - 33),[4] to which no objections have been advanced. In the case of two of the changes recommended last year, Subcommittee I on Standard Specifications for Fire Tests of Materials and Construction (G. N. Thompson, chairman) has recommended further modifications. On the recommendation of this subcommittee, Committee C-5 recommends that the tentative revision of this standard, with the modifications as given below, be approved for reference to letter ballot of the Society for adoption as standard:

Table I.—Change the proposed revised table to read as shown in the accompanying Table I by the addition of the italicized words and figures and the omission of those in brackets.

Change the heading preceding Section 22 to read as follows by the addition of the italicized words and the omission of those in brackets:

> Tests of [Finish] *Protection* for Combustible Framing, or *for Combustible* Facings *on the Unexposed Side* of Walls, Partitions, and [Ceilings] *Floors.*

Section 22.—In place of the proposed revision of this section substitute the following:

> 22. Test panels carrying wall, partition, or floor protection shall be finished with the protections which are the subject of the test, except that where the finish on the unexposed side is not the subject of the test and is not specifically indicated, the testing authority shall apply a finish judged suitable for the purpose. In case a floor construction as installed for actual use is to have no finish on the unexposed side, it shall be so tested.

TABLE I.

Resistance Period	Water Pressure at Base of Nozzle, psi.	Duration of Application, min. per 100 sq. ft. exposed area
8 hr. and over	45	6
4 hr. and over if less than 8 hr.	45	5
2 hr. and over if less than 4 hr.	30	2½
[1 hr.] *1 1/2 hr.* and over if less than 2 hr.	30	1½
[Less than] 1 hr. *and over if less than 1 1/2 hr.* (if desired)	30	1

IV. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

Revisions in the Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring (C 132 - 40 T) were proposed and accepted at the 1940 annual meeting. Subcommittee IV on Fire Tests of Scaffolding (W. B. White, chairman) reported at the meeting of Committee C-5 on March 5, 1941, that no comments or criticisms of the tentative standard had been received and recommended that the specifications be submitted to the Society at the annual meeting for adoption as standard. At the meeting of the main committee, however, it was voted that the specifications be continued as tentative for another year on the ground that the small difference in weight loss in the timber test between well-treated and untreated material, making it necessary to set the limit (30 per cent) at a figure that can be met by untreated wood, made the criterion of acceptance of doubtful value, and on the further ground that other methods of testing might be adapted to advantage for the purposes of the specifications.

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[2] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 194.

[3] 1940 Supplement to Book of A.S.T.M. Standards Part II, p. 338.

[4] 1939 Bcok of A.S.T.M. Standards, Part II, p. 152.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 32 members; 26 members returned their ballots, with the results shown in Table II.

This report has been submitted to letter ballot of the committee which consists of 32 members; 24 members returned their ballots, all of whom have voted affirmatively.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standard			
Test for Fire-Retardant Properties of Wood	22	1	3
II. Adoption of Tentative Standard as Standard			
Fire Tests of Door Assemblies (C 152 - 40 T)	24	2	0
III. Adoption as Standard of Tentative Revision			
Spec. for Fire Tests of Building Construction and Materials (C 19 - 33; ASA A 2-1934), as revised	24	1	1

Respectfully submitted on behalf of the committee,

R. P. Miller,
Chairman.

H. M. Robinson,
Secretary.

REPORT ON COMPARATIVE FIRE TESTS OF TREATED AND UNTREATED WOOD

REPORT OF SUBCOMMITTEE II ON FIRE TESTS OF LUMBER

INTRODUCTION

The scope of activities assigned to this subcommittee by Committee C-5 according to the minutes of the meeting of July 3, 1936, is as follows:

> The preparation of specifications governing fire tests to determine the fire hazard of combustible materials, both natural and treated to render them more fire-retardant, applicable to materials used in the interior finish of buildings and not to those elements on which the stability of the building is dependent; not to be limited to materials treated by any one specified process, but to include test methods applicable to untreated lumber as well as to lumber and other materials treated by impregnation, surface application, or other means of diminishing the fire hazard of the natural material.

The subcommittee has confined its present attention to test procedures for the determinination of the effectiveness of fire-retarding treatments of lumber by impregnation. While certain tests for this purpose have been employed and recommended, the data available to the committee was inconclusive for the formulation of a standard procedure. At the 1937 meeting, this subcommittee agreed to conduct a series of comparative tests to include various procedures similarly conducted by different laboratories. In thus acquiring reliable data, the primary purpose was the comparison of (*1*) the sensitivity of the test methods to different degrees of chemical treatment applied to different species of wood, and (*2*) the reproducibility of results obtained by different laboratories on material receiving similar treatment.

Five laboratories cooperated in the test program: namely,

Forest Products Laboratory,
National Bureau of Standards,
Wood Preserving Corporation,
Protexol Corporation, and
Civil Engineering Research Laboratories of Columbia University.

The materials subjected to test were furnished by the National Lumber Manufacturers Association.

OUTLINE OF TESTS

The test methods included in this investigation were as follows:

1. New York City "Crib Test,"
2. New York City "Timber Test,"
3. "Fire Tube Test" developed by the Forest Products Laboratory,
4. "Special Crib Test" developed by the Columbia University laboratories, and
5. "Flame Spread Test" developed by the National Bureau of Standards.

The New York City "crib" and "timber" tests were conducted essentially as prescribed in the New York City Building Code. These tests have been applied for over 30 yr. for the approval of impregnated fire-retardant wood used primarily as interior trim. The fire tube test[1] has been used by the Forest Products Laboratory, National Bureau of

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[1] "A New Test for Measuring the Fire Resistance of Wood," by T. R. Truax and C. A. Harrison, *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 973 (1929).

Standards, and other agencies and in 1930 was published as information by the A.S.T.M. as a Proposed Method of Test for Fire-Retardant Treated Wood.[2] The "special" crib test was given this designation to differentiate this new form of crib test from that previously used.

Complete instructions for the conduct of each of the first four tests enumerated above were furnished each of the cooperating laboratories. The flame spread tests were conducted by one of the laboratories as a supplementary series. Only a brief outline of the various test procedures will be given here to indicate the type of apparatus, specimen, procedure, and data recorded. Photographs of the essential apparatus are shown in Figs. 1, 2, and 3.

New York Crib Test:

Specimen.—Twelve pieces, ½ by ½ by 6 in.

Apparatus.—A ring stand support with a 4-in. diameter ring supporting a flat wire screen, 7 in. square, composed of ½-in. mesh made of No. 18 gage wire. A Tirrill or common bunsen burner, ⅜ in. in inside diameter, protected from drafts by a cylindrical, No. 24 gage sheet iron sleeve, 3 in. in diameter and 6 in. in height, attached to the burner with its top approximately 5¼ in. above the burner orifice.

Procedure.—Tests were made under a furnace hood to avoid drafts. The burner with the sleeve in place is adjusted to give a flame approximately 7½ in. in height and having a temperature of 1700 ± 25 F. at its hottest point as measured by a No. 18 gage chromel-alumel thermocouple. The twelve test pieces, previously weighed, are placed on the wire screen in three tiers, each containing four pieces, spaced ½ in. apart with the pieces of each tier placed perpendicular to those below it, thus forming a crib approximately centered over the supporting ring. The pieces forming the crib are placed at random with respect to surface condition. The burner with sleeve in place is so placed that the orifice of the burner tube is 6 in. below and directly under the center of the crib. The burner flame is applied for a period of 2 min.

Observations.—Observations are made of the following: maximum height of flame, duration of flame after removal of burner, duration of glow after flaming has ceased, and weight of crib remaining, including charcoal, after all flame and glow has ceased. Percentage loss in weight is computed.

Timber Test:

Specimen.—Two pieces ¾ by 1½ by 10 in.

Apparatus.—A crucible gas furnace with opening 6 in. in diameter, substantially similar to that described in the Tentative Specifications for Fire-Retardant Properties of Wood for Scaffolding and Shoring (A.S.T.M Designation: C 132 – 37 T) of the American Society for Testing Materials.[3]

Procedure.—The temperature of the furnace is measured by a No. 8 gage chromel-alumel thermocouple placed with its junction at the center of the furnace opening and 1 in. below the plane of the top surface on which the specimens are laid. After ignition of the gas, the furnace opening is wholly covered by two fire bricks and a third brick is laid across the rear end to serve as a stop for the end of the test specimen when placed over the crucible opening. The furnace is maintained at a temperature of 1700 ± 25 F. with a minimum amount of gas in the air-gas mixture used. After a stable furnace condition is reached, the covering bricks are separated and the two specimens laid flatwise, side by side, across the furnace opening. The specimens are placed snugly together and the flanking brick moved up closely so as to completely cover the furnace opening. Prior to the test the specimens are weighed and sectional dimensions measured. After 2-min. exposure the specimens are carefully withdrawn and placed in a parallel position with at least 6 in. clear space between them and with burned faces upward, across angle iron supports. This placement should be under a fume hood to avoid side drafts.

Observations.—Observations are made of the duration of flame and of glow after flaming has ceased. After glow has ceased the specimens including charcoal are weighed and percentage loss in weight computed. The specimens are then scraped free of charcoal with a dull knife, cut square across at the point of greatest sectional reduction and the remaining sectional area measured. The percentage reduction of area is computed.

Fire-Tube Test:

Specimen.—One piece, ⅜ by ¾ by 40 in.

Apparatus.—Details of the apparatus are given in the cited reference.[1] Essentially it

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 463 (1930).

[3] 1939 Book of A.S.T.M. Standards, Part II, p. 951.

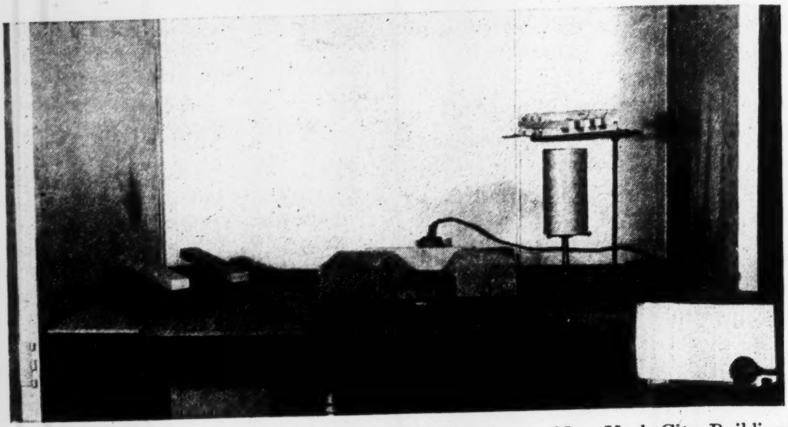


FIG. 1.—Timber Test and Crib Test Apparatus Conforming to New York City Building Code.

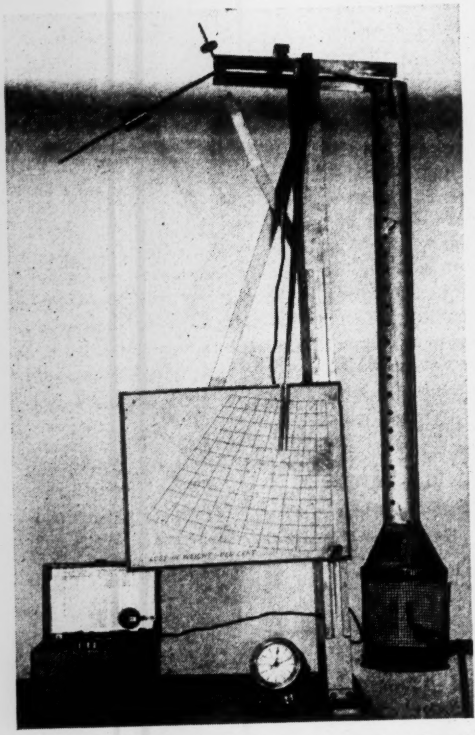


FIG. 2.—Fire Tube Test Apparatus Developed by Forest Products Laboratory.

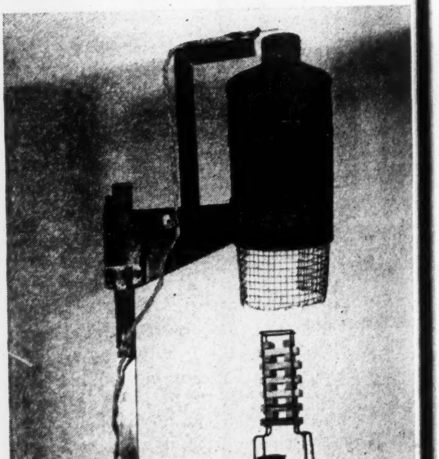


FIG. 3.—"Special" Crib Test Apparatus Developed by Columbia University Research Laboratory.

consists of a galvanized iron tube 3 in. in diameter and 38 in. in length, with $\frac{3}{8}$ in. holes in the side throughout its length, and having a cage bottom. This tube is suspended on knife edges from a weighing beam provided with a pointer which moves across a chart as the weight of the specimen changes. By suitable adjustment, the pointer will swing to the zero line of the chart when the test specimen is in place. With loss in weight of the specimen during test, readings of the pointer on the chart give the percentage loss in weight. The specimen, provided with a small hook at one end, is suspended within the tube. A No. 18 gage thermocouple is attached to the weighing beam so that the hot junction is 1 in. above, and directly over, the center of the tube. A low-form bunsen burner provides the ignition flame which is applied to the lower end of the suspended specimen.

Procedure.—After checking the adjustment of the apparatus and properly balancing, the previously weighed specimen is suspended in the tube and the chart adjusted to zero reading. The gas burner is adjusted to give a blue flame 11 ± $\frac{1}{2}$ in. in height with a tall indistinct inner cone and a temperature of 1000 ± 25 C. at the hottest point of the flame and 180 ± 5 C. at the center of the top of the tube. These temperatures are measured with a No. 18 gage thermocouple. The burner is placed with its top 1 in. below the bottom of the specimen and directly beneath it. The gas flame will envelop the lower end of the specimen and, if the specimen warps, the flame is moved by the operator so that it plays directly on the specimen. The burner flame is removed after 4 min.

Observations.—The percentage loss in weight and top temperature are recorded at intervals of 30 sec. until 2 min. after flaming stops. Observations are made of the following: maximum loss in weight, loss of weight when flaming ceases, maximum temperature, duration of flame, and duration of glow of suspended specimen or charcoal retained in cage at bottom of tube. Data computed from these observations include the area under the time-temperature curve (above the 180 C. line), and maximum rate of weight loss in percentage per minute.

"Special" Crib Test:

Specimen.—24 pieces, $\frac{1}{2}$ by $\frac{1}{2}$ by 3 in.

Apparatus.—The equipment consists of a specially designed percentage-loss scale having a scale pan provided with a permanently fixed wire frame for holding the crib specimen and a movable cylindrical shield which surrounds the specimen during test. A Meker burner provides the exposure flame. The weighing scale is self-contained in a case $3\frac{1}{4}$ by 18 by 12 in. with a semicircular aperture in one side through which the chart and moving hand may be viewed. The reading of the hand on the chart is similar to an ordinary meter or dial indicator. The chart is adjustable, by means of a knob on the face of the case, for zero reading and, as designed for these tests, is capable of accommodating specimens ranging from 110 to 280 g., original weight. An adjusting knob is provided for no-load position of the indicating hand and a lock to prevent oscillation of the scale when not in use. A scale pan, 7 in. in diameter, over the top of the scale case supports a permanently attached wire frame. The four vertical legs of this frame are spaced about 4 in. apart near the bottom and 2 in. apart in the upper section. Horizontal wires are attached to the legs at the top and bottom of the narrower section of the frame. The wider space within the lower part of the frame permits a burner to be placed directly under the crib specimen which is placed in 12 tiers of two pieces each on the transverse wires provided. This frame facilitates the spacing of the pieces and duplication of specimen placement as well as maintaining an easily reproducible position of the specimen with reference to the burner flame. A Meker burner with a grid 2 in. in diameter is attached to a swinging arm with bearing support attached to the shield supporting stand. This burner can be adjusted for elevation and a stop latch on the swinging arm support can be adjusted so that the burner, when moved forward, will be exactly centered under the crib specimen and a definite distance below it. Removal of the burner is accomplished by swinging the arm outward. The cylindrical shield, 8 in. in diameter by 12 in. in height, can be moved vertically on a guide bar support attached to the bench, adjacent to the weighing scale. A conical top with 3-in. opening is attached to the cylindrical section and a thermocouple is permanently mounted $\frac{1}{2}$ in. above this top opening. The front of the cylindrical section of the shield is provided with a mica observation window. This shield is placed symmetrically around the test specimen in the wire frame at a constant elevation.

Procedure.—The test pieces taken at random are placed in the wire frame in 12 tiers, each containing two pieces 1 in. apart with pieces in each tier placed perpendicular to those below. The first three tiers are placed on transverse supporting wires. The spacing of pieces in the crib is duplicated by placing each piece in contact with the vertical wires of the frame, permitting approximately equal overhang at the ends. The weighing scale is set to zero reading as required by the initial weight of the specimen. The Meker burner is adjusted to produce a blue flame 10 ± ½ in. in height having a temperature of 1800 ± 25 F. at a point 1½ in. above the grid, as measured with a No. 18 gage chromel-alumel thermocouple. The temperature at the top of the shield is then 600 ± 15 F. after a short period of warming. The burner, previously adjusted so that the grid is 1½ in. below the crib, is swung into position, thus applying the flame to the specimen without further manipulation. The flame is applied for 3 min.

Observations.—Percentage loss in weight and top temperature are recorded at intervals of 30 sec. until successive readings show a weight loss of less than 0.5 per cent (or until glow has ceased); readings are taken to 0.5 per cent or less. Observations are made of the following: maximum weight loss, loss of weight when flaming ceases, maximum temperature, duration of flame, and duration of glow of remaining crib or of such pieces as may fall into the scale pan. Computed data include the area under the time-temperature curve (above the 600 F. line), and the maximum rate of weight loss in percentage per minute.

Flame Spread Test:

This test was conducted by laboratory F only, as a series supplementing the other comparative tests.

Specimen.—One piece, ¼ by ¾ by 18 in.

Apparatus.—A ring stand support suitable for suspending the 18-in. specimen vertically over a ⅜-in. Tirrill burner. Tests are made in a room free from drafts or with an open-front shield around the specimen.

Procedure.—The specimen is suspended in a vertical position with its lower end 2 in. above the top of the ⅜-in. burner, regulated to give a flame having a small inner cone and producing 3000 Btu. per hr. The flame is applied for 1 min.

Observations.—Observations are made of the following: loss in weight by weighing before and after test, maximum height of flame, duration of flame and glow.

MATERIALS

In order to afford the means of comparing the sensitivity of the various test methods, it was desirable to include species of wood that would provide a range in density and susceptibility to treatment. In view of the large amount of data to be obtained, the program was limited to three species: namely, southern yellow pine (sapwood), white oak (heartwood), and yellow poplar (heartwood). These species differ in density and natural resistance to combustion. While the pine was expected to take uniform treatment, some variations were probable in the poplar, and also the oak, which is known to be difficult to treat thoroughly and was expected to have only partial retention of treating chemical.

In the selection of type of treatment, it was not intended to compare the effectiveness of different formulas. For the purpose of these tests, solutions of ammonium salts (ammonium sulfate, 80 per cent, and monoammonium phosphate, 20 per cent) were used for all treatments except for one series of pine specimens which was treated with a solution of borax (100 per cent). The schedule of treatment with various concentrations of the above salts was as follows (see also Table I):

Species	Concentrations of Treatments, per cent	
	Ammonium Sulfate, 80 per cent and Monoammonium Phosphate, 20 per cent	Borax, 100 per cent
Southern yellow pine (sapwood)	0, 2.5, 7.5, 12.5	7.5
Yellow poplar (heartwood)	0, 5, 12.5	...
White oak (heartwood)	0, 12.5	...

The inclusion of one series treated with borax was intended to provide another means of comparing the different test methods, since this salt is not effective in preventing continued glow after flaming has ceased.

After treatment of the boards from which test specimens were prepared, samples were cut transversely from each board for examination of distribution of fire retardant. Quantitative determination of distribution was not attempted.

From the discoloration produced by baking the specimens containing ammonium salts, the 12.5 per cent concentration of solution appeared to distribute the fire-retardant salts rather uniformly throughout the pine and poplar boards, but little beyond the surface of the oak boards.

TABLE I.—TREATMENTS OF TEST BOARDS.

NOTE 1.—Test boards were treated with solutions of ammonium salts (ammonium sulfate, 80 per cent, monoammonium phosphate, 20 per cent) of the concentrations shown; except that the 5.6 per cent concentration represents treatment with a solution of anhydrous borax in that percentage by weight.

NOTE 2.—Absorption values are expressed as the weight in pounds of dry salt retained after treatment per cubic foot of wood.

NOTE 3.—Specific gravity is based on the air-dry weight (65 per cent relative humidity at 80 F.) and the air-dry volume.

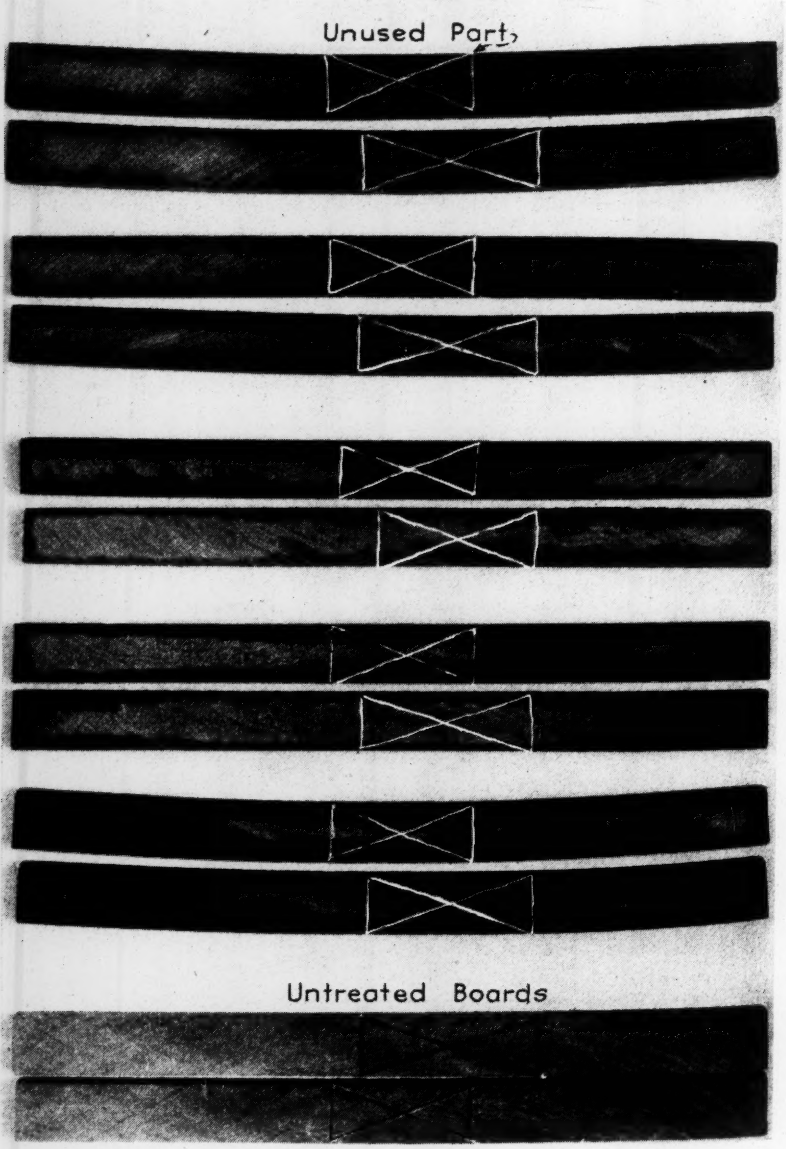
SOUTHERN YELLOW PINE, SAPWOOD

Laboratory	Untreated		2.5% Concentration			7.5% Concentration			12.5% Concentration			5.6% Concentration		
	Board No.	Sp. Gr.	Board No.	Sp. Gr.	Absorption	Board No.	Sp. Gr.	Absorption	Board No.	Sp. Gr.	Absorption	Board No.	Sp. Gr.	Absorption
F	39-A	0.503	2-A	0.600	1.06	10-A	0.556	3.35	9-A	0.554	5.71	1-A	0.557	2.48
	39-B	0.545	2-B	0.579	1.07	10-B	0.574	3.32	9-B	0.566	5.65	1-B	0.545	2.53
P	46-A	0.546	29-A	0.572	1.07	15-A	0.535	3.30	13-A	0.604	5.51	20-A	0.549	2.57
	46-B	0.508	29-B	0.599	1.06	15-B	0.553	3.24	13-B	0.594	5.66	20-B	0.525	2.62
W	48-A	0.541	44-A	0.598	1.07	19-A	0.561	3.23	30-A	0.581	5.62	31-A	0.572	2.48
	48-B	0.505	44-B	0.584	1.08	19-B	0.537	3.27	30-B	0.597	5.53	31-B	0.563	2.52
C	52-A	0.542	50-A	0.573	1.06	26-A	0.536	3.32	32-A	0.574	5.58	35-A	0.571	2.41
	52-B	0.590	50-B	0.573	1.07	26-B	0.553	3.29	32-B	0.593	5.60	35-B	0.552	2.46
B	56-A	0.606	53-A	0.590	1.06	28-A	0.556	3.28	36-A	0.561	5.75	47-A	0.551	2.46
	56-B	0.600	53-B	0.560	1.09	28-B	0.565	3.26	36-B	0.583	5.60	47-B	0.542	2.52

Laboratory	YELLOW POPLAR, HEARTWOOD								WHITE OAK, HEARTWOOD				
	Untreated		5.0% Concentration			12.5% Concentration			Untreated		12.5% Concentration		
	Board No.	Sp. Gr.	Board No.	Sp. Gr.	Absorption	Board No.	Sp. Gr.	Absorption	Board No.	Sp. Gr.	Board No.	Sp. Gr.	Absorption
F	3-A	0.488	4-A	0.501	2.30	8-A	0.479	6.25	5-A	0.685	11-A	0.807	1.45
	3-B	0.467	4-B	0.514	2.27	8-B	0.471	6.29	5-B	0.694	11-B	0.794	1.60
P	6-A	0.463	9-A	0.503	2.27	10-A	0.426	6.14	12-A	0.808	16-A	0.693	1.73
	6-B	0.481	9-B	0.488	2.22	10-B	0.419	6.24	12-B	0.706	16-B	0.745	1.67
W	7-A	0.465	14-A	0.444	1.60	13-A	0.420	6.43	22-A	0.809	17-A	0.712	2.09
	7-B	0.484	14-B	0.448	1.49	13-B	0.408	6.50	22-B	0.783	17-B	0.727	2.01
C	21-A	0.482	16-A	0.518	1.39	23-A	0.413	6.38	23-A	0.735	18-A	0.674	1.43
	21-B	0.489	16-B	0.536	1.23	23-B	0.417	6.45	23-B	0.725	18-B	0.701	1.36
B	25-A	0.548	17-A	0.482	1.95	24-A	0.423	6.22	24-A	0.783	21-A	0.647	1.50
	25-B	0.540	17-B	0.486	2.11	24-B	0.424	6.25	24-B	0.747	21-B	0.645	1.49

The 7.5 per cent concentration of solution in pine afforded a distribution less uniform than the 12.5 per cent but much better than the 2.5 per cent in pine or the 5 per cent in the poplar where there was apparently little retardant in the central part of the boards. Figures 4 and 5 show the distribution obtained in yellow poplar boards treated with a 5 per cent concentration of solution and in white oak boards treated with a 12.5 per cent solution.

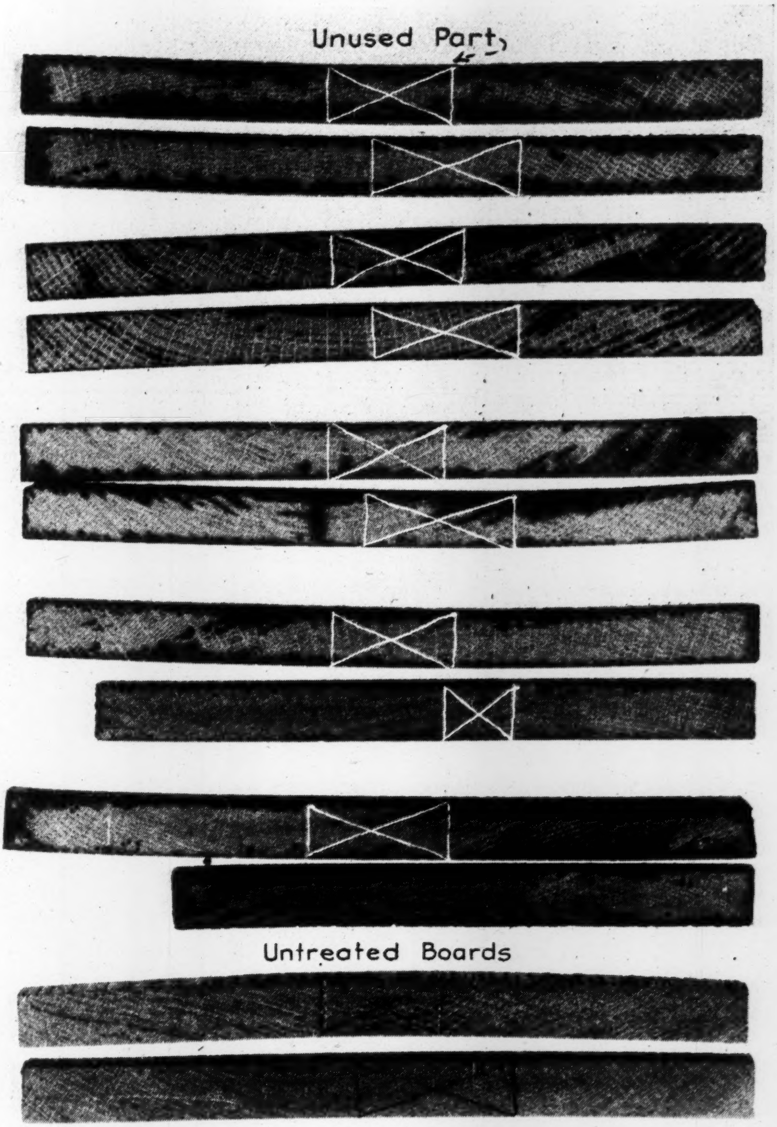
Inasmuch as each of the five laboratories conducted four different types of test (plus an additional series by laboratory F) for each concentration of treatment, it was obviously impossible to obtain all test specimens for a single board. This necessitated care in the selection of boards so that the five pine boards, three poplar boards, and two oak boards from which test specimens were prepared for any one laboratory would (*a*) Boards treated with a 5 per cent solution of ammonium sulfate, 80 per cent, and monoammonium phosphate, 20 per cent.


Unused Part
Untreated Boards

(*b*) Untreated boards.

FIG. 4.—Distribution of Fire-Retardant Chemical in Yellow Poplar Boards.

(*a*) Boards treated with a 12.5 per cent solution of ammonium sulfate, 80 per cent, and monoammonium phosphate, 20 per cent.

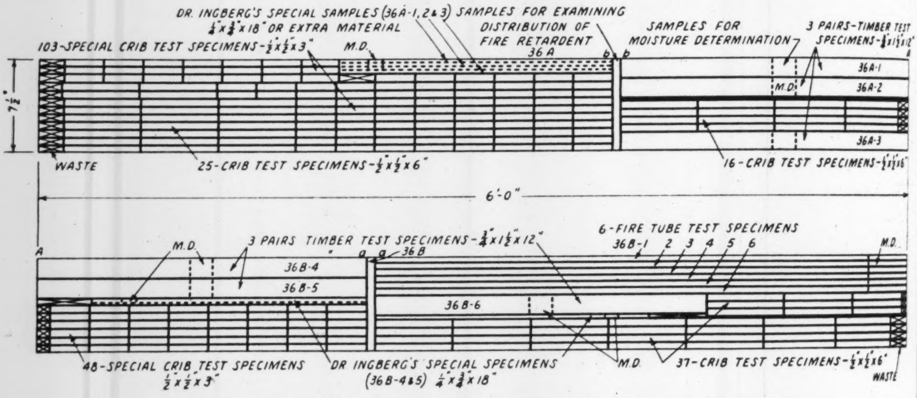

Unused Part
Untreated Boards

(*b*) Untreated boards.

FIG. 5.—Distribution of Fire-Retardant Chemical in White Oak Boards.

be closely similar in density, uniformity of treatment, etc., and also as nearly as possible the same as those used by each of the other laboratories for the same tests. This meant that 25 pine boards, 15 poplar boards, and 10 oak boards were required with similar treatment and natural characteristics. A sufficient number of boards were selected, and about twice the required number were treated so that after final selection a reasonable uniformity resulted in the specimens furnished among and within laboratories. The specific gravity and retention of retardant for each board is recorded in Table I which shows the treatments of test boards. The materials, furnished by the National Lumber Manufacturers Association, were selected, treated, and cut into the various test specimens by the Forest Products Laboratory.

As previously indicated, different test specimens were required for each of the five tests conducted. Six test specimens were prepared for each type of test from the same board. Thus, the various tests made by any one laboratory on a single treatment and species were directly comparable; while similar sets of specimens for other treatments were taken from other boards, variations due to natural characteristics and uniformity of treatment were minimized by careful selection. The stock consisted of boards $\frac{3}{4}$ in. in thickness by 12 ft. 0 in. in length, and surfaced on four sides. Oak and poplar boards were 10 in. in width, and the pine boards were $7\frac{1}{2}$ in. in width. These were cut in half, forming 6-ft. lengths, which were treated, dried, and cut into the individual pieces required. The location of the various test specimens in the original board, for all treatments, is shown on the cutting diagram, Fig. 6. Sufficient material was provided, either as extra length or extra pieces, for determination of the moisture content at the time of test. All of the specimens were ripped to width with a fine circular saw, without further planing, and all crib specimens were cut from one face with a tolerance in dimensions


DR. INGBERG'S SPECIAL SAMPLES (36A-1, 2 & 3) ¼"x¾"x18" OR EXTRA MATERIAL
SAMPLES FOR EXAMINING DISTRIBUTION OF FIRE RETARDENT
SAMPLES FOR MOISTURE DETERMINATION
3 PAIRS-TIMBER TEST SPECIMENS-¾"x1½"x12"
103-SPECIAL CRIB TEST SPECIMENS-½"x½"x3"
M.D.
36 A
b
b
A
36A-1
36A-2
36A-3
M.D.
7½"
WASTE
25-CRIB TEST SPECIMENS-½"x½"x6"
16-CRIB TEST SPECIMENS-½"x½"x6"
6'-0"
6-FIRE TUBE TEST SPECIMENS
36B-1 2 3 4 5 6
M.D.
A
M.D.
3 PAIRS TIMBER TEST SPECIMENS-¾"x1½"x12"
a
a
36 B
36B-4
36B-5
36B-6
48-SPECIAL CRIB TEST SPECIMENS ½"x½"x3"
DR INGBERG'S SPECIAL SPECIMENS (36B-4 & 5) ¼"x¾"x18"
M.D.
37-CRIB TEST SPECIMENS-½"x½"x6"
WASTE

NOTE: STOCK FOR ALL CRIB TEST SPECIMENS TO BE REDUCED TO ½" THICKNESS BY PLANING THE ENTIRE ¼" FROM ONE SURFACE.
UNUSED CRIB TEST SPECIMENS SHOULD BE USED FOR MOISTURE DETERMINATIONS.
THE ORIGINAL 12'-0" BOARD WAS CUT AT A-A.

FIG. 6.—Diagram for Cutting Fire Test Specimens from Two Halves of a Board $\frac{3}{4}$ in. by $7\frac{1}{2}$ in. by 12 ft. 0 in. in dimensions.
(Showing location of marking on the unsurfaced side of board No. 36.)

of less than $\frac{1}{32}$ in. All samples were conditioned in the Forest Products Laboratory at a temperature of 80 F. in an atmosphere having a relative humidity of 30 per cent, resulting in a moisture content of approximately 7 per cent in the untreated wood and slightly higher in the treated samples, due to the presence of the absorbed salts. The cut specimens, suitably bundled and marked, were wrapped in waterproof paper for shipment to the respective laboratories. It was hoped that all materials brought to equilibrium by the above conditioning would not vary in moisture content when treated by more than plus or minus 3 per cent. The actual test records show that the moisture content was generally within these limits. As a check on changes in moisture, each bundle of specimens was weighed before leaving the Forest Products Laboratory so that each testing laboratory could determine the condition of the sample before test.

It should be noted that all reasonable precautions were taken to provide uniform materials for these tests. Absolute uniformity in a material such as wood cannot be expected, and some variations in the retention of retardant chemical have been noted. The sensitivity of the various test methods in detecting such variations is indicated by the test results.

TEST DATA

In conducting each of the tests, six specimens were tested. One of these specimens, according to the program, was used as a practice specimen, and the data from the remaining five were reported for the record ($n = 5$). The large number of individual observations recorded will not be presented here. Tables II to XXIII give the maximum, and minimum values, also the average, $\bar{X}$, and the standard deviation, σ, for each set of specimens as determined by each of the testing laboratories. The several laboratories are designated as laboratories C, F, W, B, and P. The board numbers, specific gravity, moisture content of the specimens before test, and retention of retardant chemical are shown. The grand average $\bar{\bar{X}}$ of each variant, the average standard deviation, $\bar{\sigma}$, and the coefficient of variation, v, are included in the summary of data. These tables include the essential data required for analysis.

ANALYSIS OF DATA

An examination of the data indicates that the results obtained by the different laboratories vary considerably for some of the treatments and species. On the whole, the reproducibility within samples taken from the same board and under one test condition is better than between laboratories. Before making comparisons of test methods, it was necessary to determine the extent of variations in the results for each test. A statistical analysis was made of some of the data for evidence of "control" that is, information indicating whether the variations between observations are due to "chance" causes or whether other assignable causes are responsible. Such analyses were made of the average values of final percentage of weight loss obtained in the four tests conducted by all laboratories and the maximum temperature observed by all laboratories in the fire-tube and "special" crib tests. These two observations are the most dependable and serve to indicate the extent of variations which may be due to the samples or test method.

In the study of the variability of the individual observations and the averages of the various samples, two functions of the data containing the essential information have been employed: namely, the average, $\bar{X}$, and the standard deviation, σ. The average, $\bar{X}$, is the arithmetic

TABLE II.—FINAL WEIGHT LOSS IN FIRE TUBE TESTS.

NOTE 1.—Values for final weight loss are given as percentages of the weight of the specimen.

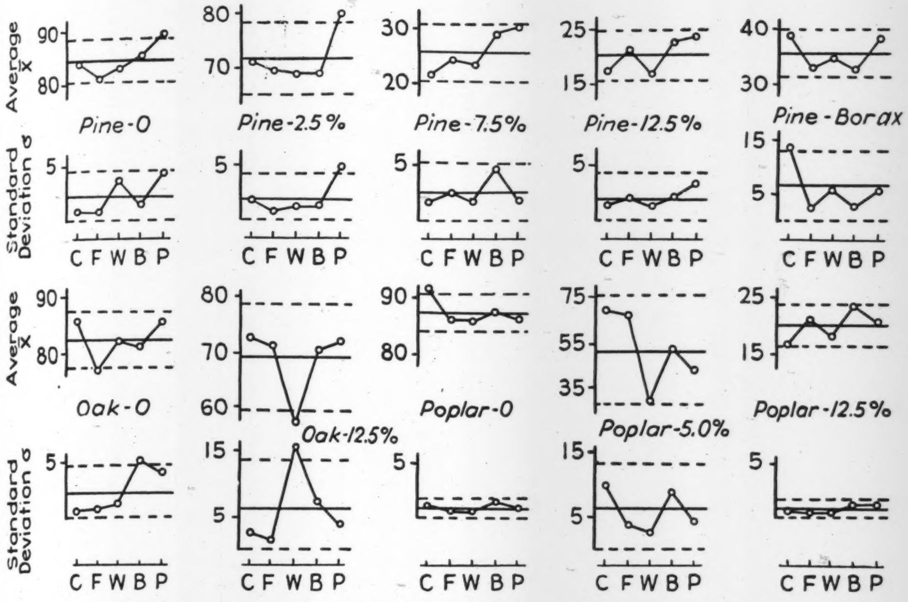
NOTE 2.—For information on treatments of test boards, see Table I.

NOTE 3.—Absorption values are expressed as the weight in pounds of dry salt retained after treatment per cubic foot of wood.

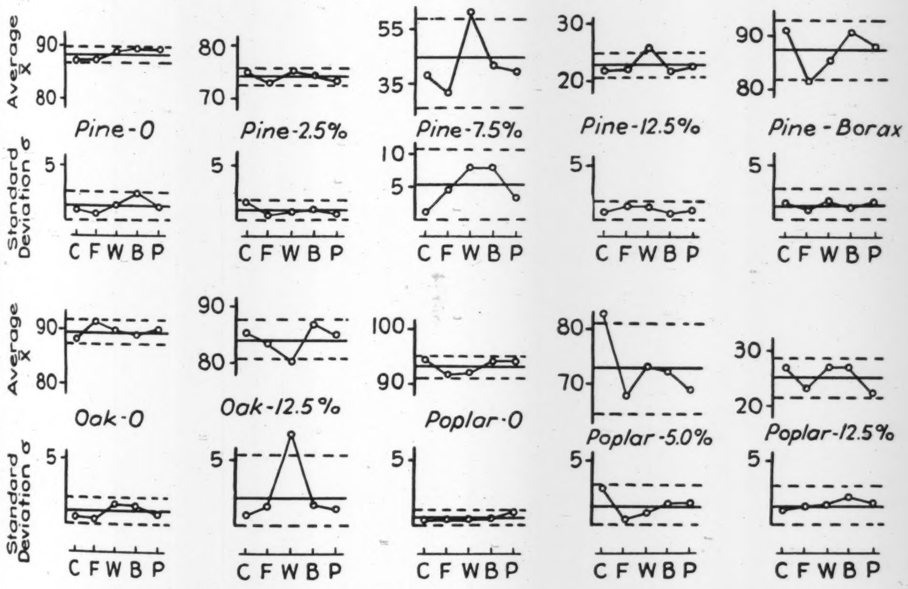
NOTE 4.—Specific gravity is based on the air-dry weight (65 per cent relative humidity at 80 F.) and the air-dry volume.

NOTE 5.—The symbols used are as follows: $\bar{X}$ = average, σ = standard deviation, and v = coefficient of variation. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Board No.	52	39	48	56	46		23	5	22	24	12	
Sp. gr.	0.590	0.524	0.505	0.600	0.508	0.545	0.725	0.690	0.783	0.747	0.706	0.730
Moisture, per cent	6.8	6.2	8.1	5.6	8.95	7.13	7.2	7.3	7.5	8.0	9.68	0.794
Absorption	0	0	0	0	0	0	0	0	0	0	0	0
Weight loss, per cent:												
Max.	85.0	82.0	87.0	87.0	94.0	87.00	86.0	78.0	83.0	91.0	91.0	85.80
Min.	83.0	80.0	77.5	83.0	82.0	81.10	85.0	76.0	80.0	77.0	81.0	79.80
$\bar{X}$	84.1	81.4	83.0	85.0	89.0	84.50	85.7	77.2	82.2	81.2	85.4	82.34
σ	0.80	0.80	3.62	1.41	4.19	2.165	0.40	0.75	1.17	5.08	4.08	2.295
v	0.95	0.98	4.36	1.66	4.71	2.56	0.47	0.97	1.42	6.26	4.78	2.79
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Board No.	50	2	44	53	29		18	11	17	21	16	
Sp. gr.	0.573	0.590	0.584	0.560	0.599	0.581	0.701	0.800	0.727	0.645	0.745	0.723
Moisture, per cent	8.2	6.7	9.0	7.3	9.68	8.18	5.7	7.5	7.4	7.8	9.12	7.50
Absorption	1.07	1.06	1.08	1.09	1.07	1.074	1.36	1.52	2.01	1.49	1.73	1.622
Weight loss, per cent:												
Max.	73.0	70.0	70.5	70.0	88.0	74.30	76.0	73.0	69.0	78.0	77.0	74.60
Min.	68.0	68.0	68.0	67.0	75.0	69.20	69.0	70.0	29.0	46.0	66.0	56.00
$\bar{X}$	71.7	69.6	68.9	68.8	80.0	71.80	72.9	71.8	57.4	70.6	72.2	68.98
σ	1.89	0.80	1.11	1.17	4.88	1.969	2.42	1.17	15.40	7.20	3.97	6.032
v	2.64	1.15	1.61	1.70	6.10	2.74	3.32	1.63	26.83	10.20	5.50	8.74
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Board No.	26	10	19	28	15		21	3	7	25	6	
Sp. gr.	0.553	0.565	0.537	0.565	0.553	0.554	0.489	0.477	0.484	0.540	0.481	0.049
Moisture, per cent	9.2	7.0	7.8	8.65	6.54	7.84	5.5	5.7	7.3	6.1	8.89	6.70
Absorption	3.29	3.33	3.20	3.26	3.30	3.276	0	0	0	0	0	0
Weight loss, per cent:												
Max.	24.0	27.0	25.0	34.0	33.0	28.60	93.0	87.0	86.5	90.0	87.0	88.70
Min.	19.0	20.0	20.0	21.0	28.0	21.60	90.0	85.0	85.5	86.0	85.0	84.30
$\bar{X}$	21.8	24.8	23.6	29.0	30.4	25.92	91.6	86.0	85.9	87.4	86.0	87.38
σ	1.81	2.56	1.85	4.47	1.86	2.510	1.08	0.63	0.37	1.50	0.89	0.893
v	8.30	10.32	10.32	15.41	6.12	9.684	1.18	0.73	0.43	1.72	1.03	1.02
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Board No.	32	9	30	36	13		16	4	14	17	9	
Sp. gr.	0.593	0.560	0.597	0.583	0.594	0.585	0.536	0.507	0.448	0.486	0.488	0.493
Moisture, per cent	10.5	6.9	7.9	8.4	8.99	8.54	6.60	6.20	7.70	7.15	10.40	7.61
Absorption	5.60	5.68	5.53	5.60	5.51	5.584	1.23	2.28	1.49	2.11	2.27	1.876
Weight loss, per cent:												
Max.	19.5	24.0	17.0	25.0	27.0	22.50	75.0	69.0	30.0	63.0	51.0	57.60
Min.	16.0	18.0	13.5	19.0	20.0	17.30	48.5	59.0	22.5	41.0	37.0	41.60
$\bar{X}$	17.4	21.0	15.9	22.6	23.6	20.10	68.5	66.8	27.6	52.2	42.6	51.54
σ	1.36	2.00	1.28	2.06	3.14	1.968	10.26	3.92	2.75	9.28	4.63	6.168
v	7.82	9.52	8.05	9.12	13.31	9.79	15.00	5.87	9.95	17.78	10.86	11.96
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Board No.	35	1	31	47	20		23	8	13	24	10	
Sp. gr.	0.552	0.551	0.563	0.542	0.525	0.546	0.417	0.475	0.408	0.424	0.419	0.428
Moisture, per cent	12.2	9.5	10.4	10.3	9.28	10.34	7.2	6.2	6.7	6.0	11.30	7.48
Absorption	2.46	2.50	2.52	2.52	2.57	2.514	6.45	6.27	6.50	6.25	6.14	6.322
Weight loss, per cent:												
Max.	65.5	36.0	42.0	35.0	49.0	45.50	18.0	22.0	19.0	25.0	22.0	21.20
Min.	24.5	30.0	25.0	29.0	33.0	28.30	15.5	20.0	17.5	22.0	19.0	18.80
$\bar{X}$	39.0	33.2	35.0	33.0	38.6	35.76	16.8	21.0	18.2	23.6	20.8	20.08
σ	13.93	2.32	5.66	2.28	6.15	6.068	0.81	0.63	0.50	1.02	1.17	0.826
v	35.72	6.99	16.17	9.94	15.93	16.97	4.82	3.00	2.74	4.32	5.63	4.12



Control Charts—Fire Tube Tests—Final Weight Loss

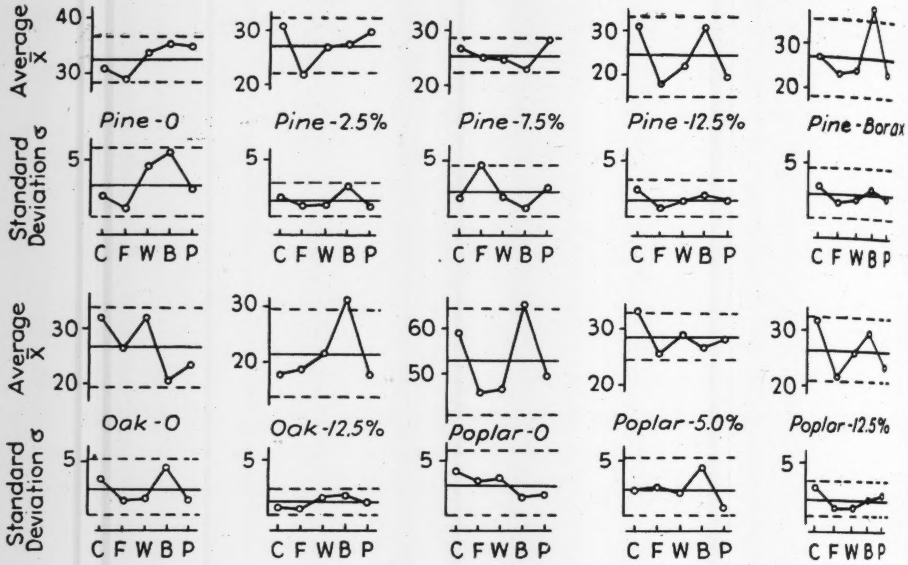


Control Charts—Special Crib Tests—Final Weight Loss

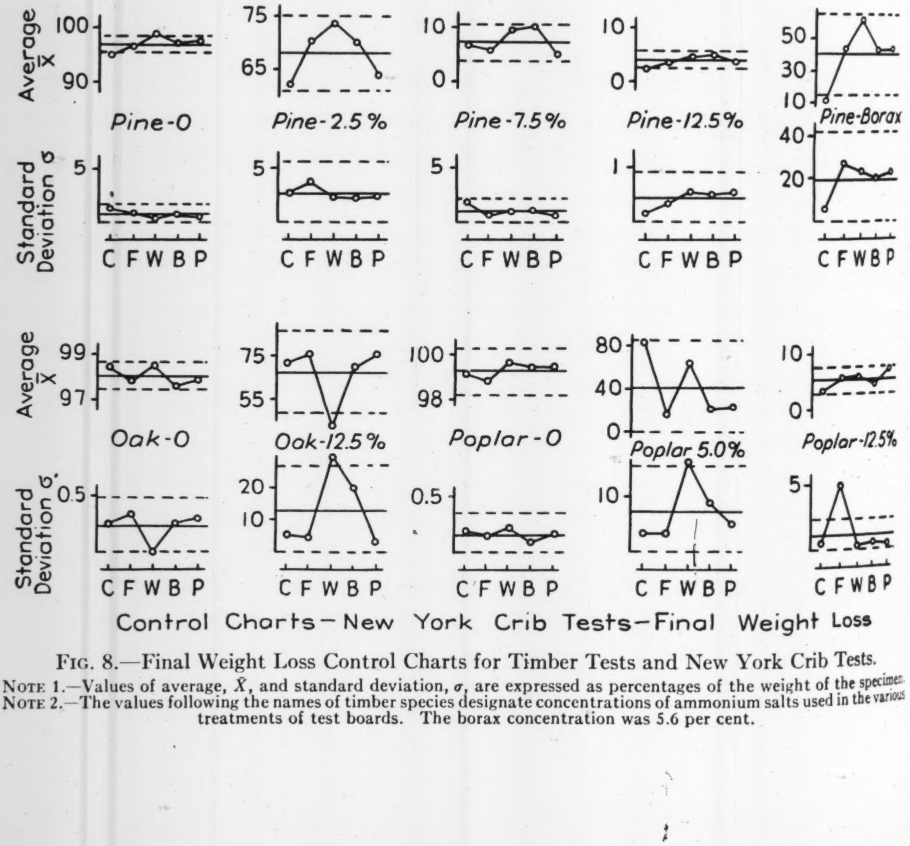
FIG. 7.—Final Weight Loss Control Charts for Fire Tube Tests and "Special" Crib Tests.

NOTE 1.—Values of average, $\bar{X}$, and standard deviation, σ, are expressed as percentages of the weight of the specimen.

NOTE 2.—The values following the names of timber species designate concentrations of ammonium salts used in the various treatments of test boards. The borax concentration was 5.6 per cent.



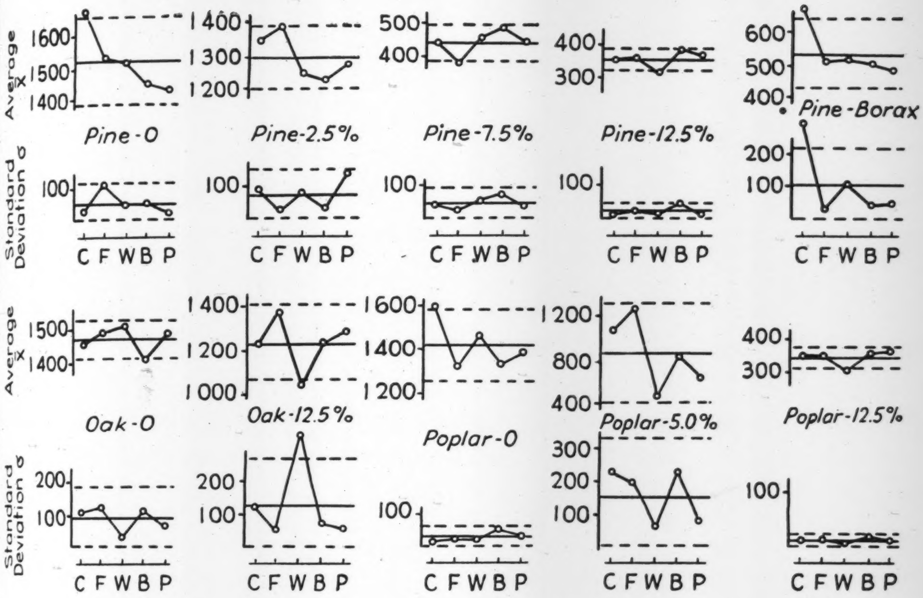
Control Charts—Timber Tests—Final Weight Loss



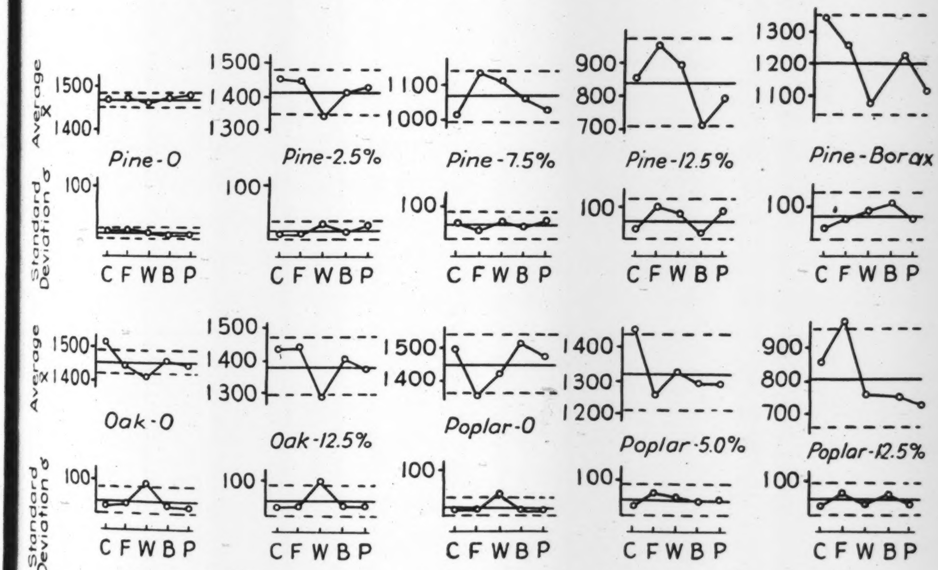
Control Charts—New York Crib Tests—Final Weight Loss

FIG. 8.—Final Weight Loss Control Charts for Timber Tests and New York Crib Tests.

NOTE 1.—Values of average, $\bar{X}$, and standard deviation, σ, are expressed as percentages of the weight of the specimen.
NOTE 2.—The values following the names of timber species designate concentrations of ammonium salts used in the various treatments of test boards. The borax concentration was 5.6 per cent.



Control Charts — Fire Tube Tests — Maximum Temperature



Control Charts — Special Crib Tests — Maximum Temperature

FIG. 9.—Maximum Temperature Control Charts for Fire Tube Tests and "Special" Crib Tests.

NOTE 1.—Values of average, $\bar{X}$, and standard deviation, σ, are in degrees Fahrenheit.

NOTE 2.—The values following the names of timber species designate concentrations of ammonium salts used in the various treatments of test boards. The borax concentration was 5.6 per cent.

mean of the set of observations and serves the purpose of summarizing in a single value the magnitude of the set of observations on the scale of measurement. It provides a measure of the central tendency of the observations. The standard deviation, σ, is a measure of the dispersion or scatter of the observations from the central value. The standard deviation is defined mathematically as the square root of the average of the squares of the deviations of the observations from their arithmetic mean (root-mean-square). The significance of the standard deviation is similar to that in mechanics of "the radius of gyration of a set of n equal particles, with respect to a given centroidal axis." The value of the coefficient of variation, v, shown in the summary tables represents the standard deviation expressed as a percentage of the average of the observations and serves to compare relative variability. The "range" indicated by the maximum and minimum values shown in Tables II to XXIII is another measure of dispersion.

TABLE III.—DURATION OF FLAME IN FIRE TUBE TESTS.

NOTE 1.—Values for duration of flame after removal of the burner are given in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table II. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	0.75	1.80	0.50	0.67	1.55	1.05	1.25	1.97	1.50	1.70	1.17	1.51
Min.	0.50	0.40	0	0	0	0.18	0.50	0.82	1.00	0.58	0.67	0.71
$\bar{X}$	0.55	0.70	0.30	0.45	0.69	0.54	0.95	1.36	1.30	1.26	0.90	1.15
σ	0.10	0.55	0.19	0.26	0.54	0.329	0.25	0.49	0.25	0.38	0.17	0.308
v	18.2	78.6	63.4	52.8	58.2	60.95	26.6	36.0	19.2	30.3	18.9	26.78
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	1.00	1.45	1.58	1.08	1.83	1.38	2.00	2.38	3.50	2.00	5.70	3.11
Min.	1.00	1.05	1.25	0.50	1.33	1.02	1.00	1.92	0	1.25	2.83	1.40
$\bar{X}$	1.00	1.19	1.45	0.82	1.57	1.21	1.75	2.18	1.83	1.60	4.07	2.29
σ	0	0.15	0.14	0.26	0.22	0.154	0.39	0.15	1.18	0.27	1.06	0.612
v	0	12.6	9.6	31.7	14.0	12.72	22.6	6.9	64.5	16.9	26.1	26.71
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	0	0	0	0	0	0	0	0	0	0.75	0.92	0.33
Min.	0	0	0	0	0	0	0	0	0	0.25	0.33	0.11
$\bar{X}$	0	0	0	0	0	0	0	0	0	0.46	0.58	0.21
σ	0	0	0	0	0	0	0	0	0	0.26	0.24	0.100
v	0	0	0	0	0	0	0	0	0	56.5	41.4	47.6
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	0	0	0	0	0	0	1.50	2.68	0	3.00	0	1.43
Min.	0	0	0	0	0	0	0.50	1.53	0	0	0	0.40
$\bar{X}$	0	0	0	0	0	0	1.00	1.95	0	1.04	0	0.80
σ	0	0	0	0	0	0	0.45	0.39	0	1.15	0	0.396
v	0	0	0	0	0	0	45.0	20.0	0	110.5	0	49.5
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	3.00	0	0	0.25	0	0.65	0	0	0	0.25	0	0.05
Min.	0	0	0	0	0	0	0	0	0	0	0	0
$\bar{X}$	0.60	0	0	0.05	0	0.13	0	0	0	0.05	0	0.01
σ	1.20	0	0	0.10	0	0.26	0	0	0	0.10	0	0.02
v	200.0	0	0	200.0	0	200.0	0	0	0	200.0	0	200.0

Control charts provide a convenient means of studying variations. Figures 7, 8, and 9 show control charts for weight loss and maximum temperature. The plotted points represent the observed average values, $\bar{X}$, and the standard deviations, σ, with the center line indicating the grand average $\bar{\bar{X}}$ and the average standard deviation, $\bar{\sigma}$. The upper and lower limits within which variations may be assumed to be due to chance causes were determined in accordance with the recommended procedure.[4] It should be noted that variations within such limits might ordinarily be expected for random samples from homogeneous materials. The limits are based upon the standard deviation, the number of observations ($n = 5$), and constants which, in the case of manufactured products, are determined by experience or economic factors. While the wood samples used in this investigation were selected with care, it is known that the physical properties of natural wood of any one species and specific gravity vary over a wide range. The susceptibility to treatment, other factors remaining the same, introduces further differences. It therefore does not seem reasonable to predicate control limits for results observed on different boards on the average standard deviation obtained from results on samples all taken from the same board. The final weight loss data shows that the standard deviation σ_x of the averages, $\bar{X}$, between laboratories is larger than the average standard deviation, $\bar{\sigma}$, within samples by approximately 67 per cent for the fire tube test, 56 per cent for the "special" crib test, 131 per cent for the timber test, and 87 per cent for the New York crib test. In the case

TABLE IV.—DURATION OF GLOW IN FIRE TUBE TESTS.

NOTE 1.—Values for duration of glow after flaming has ceased are given in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table II. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	13.50	2.50	21.25	...	17.25	13.625	29.00	3.50	34.50	...	24.83	22.96
Min.	8.5	1.50	7.75	...	6.0	5.938	18.0	3.0	10.5	...	10.40	10.48
$\bar{X}$	10.15	1.90	13.60	...	13.81	9.85	22.25	3.20	23.36	...	16.31	16.28
σ	1.81	0.37	5.01	...	4.08	2.819	3.82	0.25	7.94	...	6.86	4.716
v	17.8	19.5	36.8	...	29.5	28.60	17.2	7.8	34.0	...	42.1	28.97
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	0.05	0.13	0	0	0	0.036	3.75	2.00	15.5	...	19.35	10.15
Min.	0.05	0.03	0	0	0	0.016	1.00	0.07	0	...	0	0.27
$\bar{X}$	0.05	0.08	0	0	0	0.03	1.95	1.31	3.10	...	3.87	2.56
σ	0	0.033	0	0	0	0.007	1.05	0.72	6.20	...	7.73	3.926
v	0	41.3	0	0	0	23.33	53.8	55.0	200.0	...	19.97	153.3
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	0.17	0.20	0	0	0	0.074	22.0	3.5	17.0	...	15.13	14.41
Min.	0	0.05	0	0	0	0.010	10.0	2.0	8.0	...	4.47	6.12
$\bar{X}$	0.066	0.12	0	0	0	0.05	13.4	2.50	12.7	...	8.97	9.39
σ	0.062	0.066	0	0	0	0.032	4.39	0.632	2.93	...	3.75	2.925
v	93.9	55.0	0	0	0	64.0	32.8	25.5	23.1	...	41.8	31.15
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	0.17	0.13	0	0	0	0.06	3.5	0.38	0	...	0	0.97
Min.	0	0.03	0	0	0	0.01	0.03	0.07	0	...	0	0.025
$\bar{X}$	0.064	0.07	0	0	0	0.03	1.42	0.20	0	...	0	0.41
σ	0.057	0.034	0	0	0	0.023	1.70	0.10	0	...	0	0.451
v	89.0	48.6	0	0	0	76.7	119.7	50.0	0	...	0	109.8
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	24.0	10.0	26.0	...	40.42	25.11	20.33	0.07	0	...	0	0.026
Min.	13.0	3.0	18.83	...	20.02	13.71	0.033	0.02	0	...	0	0.013
$\bar{X}$	19.20	5.80	22.07	...	26.56	18.41	0.033	0.032	0	...	0	0.016
σ	4.17	2.79	2.58	...	7.44	4.244	0	0.019	0	...	0	0.005
v	21.7	48.1	11.7	...	28.0	23.07	0	59.4	0	...	0	31.25

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[4] "Control Chart" Method of Analysis and Presentation of Data, Supplement B, A.S.T.M. Manual on Presentation of Data, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1418 (1935), also available as a separate reprint, Third Printing, August, 1940, reprinted, March, 1941.

of the maximum temperature observations, these statistics are about the same for the pine and oak, but in the case of the poplar samples, the standard deviation, σ_x, of the averages $\bar{X}$, is about 140 per cent larger than the average standard deviation, $\bar{\sigma}$, within samples. The control limits to be applied in studying these data might be based on experience with the material employed, and arbitrarily assigned. The limits shown on the charts are based on the data. The limits for standard deviations are based on the average standard deviation $\bar{\sigma}$ (0 and $2.128\bar{\sigma}$ for $n = 5$) and those for average, $\bar{X}$, are based on the standard deviation of the $\bar{X}$ values, σ_x (plus or minus $1.596\sigma_x$ for $n = 5$).

These charts indicate the presence of variations in the test results due to assignable causes. To what extent these variations are due to differences in the sample, inherent characteristics of the test method, or departure from uniform procedure is difficult to differentiate; but at least some of the possible causes can be inferred from the data and reported difficulties encountered by the experimenters. Giving attention to the relative position of the plotted values and particularly the recurrence of extremely high or low points in the four tests on the same species and treatment, there does not seem to be evidence that these variations are due primarily to inherent differences in the wood. The results obtained on the untreated samples of each species show reasonable agreement

TABLE V.—MAXIMUM TEMPERATURE IN FIRE TUBE TESTS.

NOTE 1.—Values for maximum temperatures are given in degrees Fahrenheit.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table II. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average
SOUTHERN YELLOW PINE, UNTREATED						
Max.	1720	1724	1544	1515	1460	1592.6
Min.	1640	1418	1427	1358	1400	1448.6
$\bar{X}$	1670	1537	1517	1448	1430	1520.4
σ	28.28	110.70	45.50	54.67	26.83	53.20
v	1.69	7.20	3.00	3.77	1.88	3.50
SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						
Max.	1440	1436	1337	1285	1390	1377.6
Min.	1330	1355	1148	1195	1020	1209.6
$\bar{X}$	1342	1391	1251	1228	1272	1296.8
σ	94.30	26.60	75.40	31.92	135.85	72.81
v	7.02	1.91	6.02	2.60	10.68	5.62
SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						
Max.	500	410	509	543	490	490.4
Min.	380	338	365	352	390	365.0
$\bar{X}$	439	383	455	485	440	440.4
σ	40.17	25.40	48.60	68.63	37.28	44.02
v	9.15	6.63	10.68	14.16	8.46	10.00
SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						
Max.	375	392	347	473	390	395.4
Min.	340	329	302	348	350	333.8
$\bar{X}$	357	361	320	382	367	357.4
σ	12.88	21.10	17.08	46.34	11.66	21.81
v	3.61	5.84	5.34	12.13	3.18	6.10
SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						
Max.	1250	545	662	552	555	712.8
Min.	425	464	392	455	430	433.2
$\bar{X}$	669	511	518	505	491	538.8
σ	294.9	31.9	104.8	32.83	43.86	101.66
v	44.05	6.24	20.23	6.50	8.93	18.83

Laboratory	C	F	W	B	P	Average
WHITE OAK, UNTREATED						
Max.	1590	1670	1544	1515	1610	1585.8
Min.	1330	1355	1454	1212	1430	1356.2
$\bar{X}$	1456	1492	1510	1409	1478	1469.0
σ	110.2	120.1	30.3	108.04	67.35	87.20
v	7.57	8.05	2.00	7.66	4.56	5.94
WHITE OAK, 12.5% CONCENTRATION						
Max.	1415	1418	1391	1311	1380	1383.0
Min.	1030	1274	518	1101	1200	1024.6
$\bar{X}$	1237	1369	1045	1239	1292	1236.4
σ	125.25	50.20	335.3	72.05	57.41	128.04
v	10.11	3.67	32.10	5.81	4.44	10.35
YELLOW POPLAR, UNTREATED						
Max.	1615	1364	1490	1418	1440	1465.4
Min.	1580	1292	1418	1233	1350	1374.6
$\bar{X}$	1599	1321	1458	1335	1381	1418.8
σ	11.58	27.0	21.0	59.56	31.05	30.04
v	0.72	2.09	1.44	4.46	2.25	2.12
YELLOW POPLAR, 5.0% CONCENTRATION						
Max.	1300	1382	554	1212	760	1041.6
Min.	665	878	365	596	555	611.8
$\bar{X}$	1095	1261	473	831	643	860.6
σ	223.16	193.50	61.0	225.02	76.39	155.81
v	20.39	15.32	12.90	27.10	11.87	18.10
YELLOW POPLAR, 12.5% CONCENTRATION						
Max.	365	356	311	366	375	354.6
Min.	335	329	302	325	350	328.2
$\bar{X}$	348	349	307	349	358	342.2
σ	9.8	10.4	3.6	15.84	10.3	9.99
v	2.82	3.27	1.17	4.54	2.88	2.92

for all tests, and while the treated samples are from other boards, it is more likely that the variations are due to nonuniformity of treatment or to characteristics of the test.

TABLE VI.—AREA UNDER TIME-TEMPERATURE CURVE IN FIRE TUBE TESTS.

NOTE 1.—Values for area under the time-temperature curve include only that portion above the 180 C. line and are expressed as the product of the temperature in degrees Fahrenheit multiplied by the time in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table II. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
Southern Yellow Pine, Untreated							White Oak, Untreated					
Max.	3520	3356	3020	2831	3170	3179.4	3750	3820	4028	3594	3630	3764.4
Min.	3220	2932	2606	2467	3010	2847.0	2320	3360	2797	2323	2790	2718.0
$\bar{X}$	3418	3156	2774	2669	3105	3024.4	2954	3592	3317	2742	3276	3176.2
σ	107.0	153.2	153.9	134.9	61.2	122.04	537	149.2	408.1	449.9	334.2	375.68
Southern Yellow Pine, 2.5% Concentration							White Oak, 12.5% Concentration					
Max.	3650	3032	2477	2747	2830	2947.2	3220	3860	3272	3001	3435	3357.6
Min.	1800	2784	1750	2057	1785	2035.2	2150	2256	273	992	2420	1618.2
$\bar{X}$	2580	2876	2264	2292	2484	2499.2	2740	3368	1811	2408	3031	2671.6
σ	597.0	94.4	234.5	240.9	379.7	309.5	407	590.8	1109.0	719.5	355.1	636.28
Southern Yellow Pine, 7.5% Concentration							Yellow Poplar, Untreated					
Max.	158	80	158	265	205	173.2	3320	2784	2977	2347	3320	2949.6
Min.	20	0	1.8	0	75	19.4	2700	2380	2704	2214	2785	2556.6
$\bar{X}$	86.8	41.2	112.7	140.1	154.0	106.9	3050	2612	2804	2304	3026	2759.2
σ	50.7	31.0	56.9	99.4	53.3	58.3	204	162.4	94.7	52.3	188.7	140.42
Southern Yellow Pine, 12.5% Concentration							Yellow Poplar, 5.0% Concentration					
Max.	15	0	0	121	65	39.2	2300	2804	148	1500	500	1450.4
Min.	0	0	0	0	0	0	500	1384	4	387	350	525.0
$\bar{X}$	6.6	0	0	29.0	26.0	12.3	1710	2400	97	774	416	1079.4
σ	6.9	0	0	46.3	26.7	16.0	634	514.8	48.8	434.7	54.3	337.32
Southern Yellow Pine, 5.6% Concentration Borax							Yellow Poplar, 12.5% Concentration					
Max.	1750	244	356	254	365	593.6	0	0	0	24.2	45.0	13.84
Min.	350	168	50	97	100	153.0	0	0	0	0	0	0
$\bar{X}$	670	204	210	147	194	285.0	0	0	0	9.7	17.0	5.34
σ	543	28	47	58	92	153.6	0	0	0	10.0	20.9	6.18

According to reports from the various operators, laboratory P had difficulty with the maintenance of uniform pressure in the gas supply, particularly during the fire tube tests. Laboratory W used Pyrofax gas, and some compressed air was necessary to produce the burner flames specified. Laboratory F also reported variation of gas pressure. Difficulties in duplicating furnace conditions in the timber test were also reported. In the "special" crib tests the final weight loss values are affected by the termination of the test in some cases before glow had completely ceased. In this test the conditions are favorable to continued flame and glow in poorly treated material, and the additional weight loss during this period may be considerable.

Inspection of the charts show that extreme values by laboratory W, which are probably due to the gas supply, are in part confirmed by the maximum temperature. In the pine series of the fire-tube tests, laboratory P reports values all relatively high, which may be due to adjustment of the apparatus, since the maximum temperature values are not correspondingly high. The greatest spread of results appears to be for the borax treatment of the pine series, the treated oak, and the 5.0 per cent concentration treatment of the poplar. The nonuniformity of treatment provided in these specimens was previously recorded. Some variations are due to the type of specimen used. In the two types of crib tests composite specimens were used in which, particularly for the "special" crib test, a poorly treated piece anywhere in the crib would contribute to greater weight loss, flaming, etc. The fire tube and timber tests are influenced by the location of the sample in the board.

TABLE VII.—MAXIMUM RATE OF WEIGHT LOSS IN FIRE TUBE TESTS.

NOTE 1.—Values for maximum rate of weight loss are expressed as the loss of weight per minute, calculated as a percentage of the original weight of the specimen.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table II. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	36.0	34.0	40.0	40.0	34.0	36.8	32.0	28.0	26.0	34.0	36.0	31.2
Min.	32.0	32.0	34.0	30.0	30.0	31.6	29.0	24.0	26.0	28.0	28.0	27.0
$\bar{X}$	34.8	33.2	37.6	34.0	32.1	34.16	30.4	25.6	26.0	31.2	30.0	28.64
σ	1.47	0.98	2.33	4.90	1.60	2.26	1.02	1.50	0	2.04	3.10	1.53
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	32.0	28.0	26.0	36.0	28.0	30.0	27.0	24.0	30.0	26.0	22.0	25.8
Min.	26.0	22.0	24.0	22.0	24.0	23.6	20.0	20.0	14.0	22.0	14.0	18.0
$\bar{X}$	28.8	24.4	25.0	27.2	26.4	26.36	24.2	21.2	22.4	23.2	18.4	21.88
σ	2.32	1.96	0.89	4.66	1.50	2.27	2.40	1.60	5.12	1.60	2.94	2.73
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	10.0	12.0	12.0	30.0	14.0	15.6	39.0	42.0	36.0	54.0	40.0	42.2
Min.	7.0	8.0	8.0	8.0	8.0	7.8	36.0	34.0	32.0	32.0	30.0	32.8
$\bar{X}$	8.6	9.6	11.2	11.6	10.8	10.36	37.2	36.4	34.2	40.8	33.2	36.36
σ	1.02	1.50	1.60	2.33	2.04	1.70	1.17	2.94	1.33	7.44	3.49	3.27
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	7.0	8.0	6.0	10.0	14.0	9.0	31.0	26.0	15.0	20.0	26.0	23.6
Min.	5.0	6.0	6.0	8.0	8.0	6.6	18.0	16.0	8.0	14.0	16.0	14.4
$\bar{X}$	6.2	7.2	6.0	8.4	9.2	7.40	26.0	21.2	12.4	17.6	20.8	19.60
σ	0.75	0.98	0	0.80	2.40	0.99	4.47	3.25	2.42	2.33	3.25	3.14
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	15.0	16.0	18.0	20.0	28.0	19.4	10.0	8.0	8.0	8.0	8.0	8.4
Min.	10.0	12.0	12.0	12.0	12.0	11.6	6.0	6.0	6.0	6.0	6.0	6.0
$\bar{X}$	13.0	14.0	14.0	16.4	18.4	15.16	7.2	7.6	7.4	7.2	6.8	7.24
σ	1.67	1.26	2.19	3.20	5.43	2.75	1.60	0.80	0.80	0.98	0.98	1.03

Referring to the weight loss charts, the ranges between control limits for $\bar{X}$, which are based on the standard deviation σ_x of the average values, are a measure of the reproducibility between laboratories. The standard deviation, σ_x, and the ranges of the values of $\bar{X}$ are smallest for the "special" crib test. The average values of σ_x for all tests performed by each of the methods are: fire-tube test, 4.44; "special" crib test, 2.84; timber test, 4.25; and New York crib test, 6.53. The standard deviations representing variations within the set of samples tested by the same laboratory are also smallest for the "special" crib test. The average values of $\bar{\sigma}$ for all tests are: fire tube test, 3.09; "special" crib test, 1.75; timber test, 2.03; and New York crib test, 4.65. It should be noted that the larger standard deviations for the fire tube tests are in part due to the fact that specimen No. 1 taken from the outside edge of the board was more effectively treated than the other specimens, thus giving smaller weight loss, etc. For the large variations in treatment, the timber test would be similarly affected. While the average deviation is thus somewhat increased by the nature of the test sample employed, the relative values obtained by the different laboratories are not affected, since the average of the five samples was determined for all laboratories.

The coefficients of variation, v (standard deviation, σ, expressed as a percentage of $\bar{X}$) are shown on the tables of derived data. While these coefficients reflect the relative values of standard deviation previously recorded, care must be exercised in making comparisons because of the relatively different values of $\bar{X}$ corresponding to the same series of specimens. In general, the coefficients of variation are lowest for the "special" crib test.

TABLE VIII.—WEIGHT LOSS AFTER REMOVAL OF BURNER IN FIRE TUBE TESTS.

NOTE 1.—Values for weight loss after removal of the burner are expressed as percentages of the original weight of the specimens.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table II. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
Southern Yellow Pine, Untreated							White Oak, Untreated					
Max.	8.5	2.0	3.0	7.0	14.0	6.9	13.5	10.0	21.0	18.0	14.0	15.3
Min.	3.0	0	0	2.0	3.0	1.6	9.5	5.0	16.0	8.0	7.0	9.1
$\bar{X}$	5.3	1.2	1.4	4.0	7.2	3.82	11.6	8.0	19.0	13.2	10.2	12.40
σ	2.18	0.75	1.07	1.90	1.94	1.57	1.46	2.10	2.09	3.25	2.32	2.24
Southern Yellow Pine, 2.5% Concentration							White Oak, 12.5% Concentration					
Max.	9	11	14	10	26	14.0	25	22	29	25	46	29.4
Min.	4.5	8	11.5	0	11	7.0	13.5	19	2	11	31	15.3
$\bar{X}$	7.3	9.4	13.2	5.0	17.8	10.54	18.7	21.0	15.6	16.6	40.0	22.38
σ	1.57	1.02	0.93	3.70	5.15	2.48	3.70	1.10	9.20	4.63	5.51	4.83
Southern Yellow Pine, 7.5% Concentration							Yellow Poplar, Untreated					
Max.	1.0	1.0	3.0	2.0	3.0	2.0	5	2	4	5	5	4.2
Min.	0.5	0	0	1.0	0	0.3	3	0	3.5	2	0	1.7
$\bar{X}$	0.60	0.40	1.20	1.40	1.20	0.96	4.2	0.4	3.6	3.6	2.0	2.76
σ	0.20	0.49	0.98	0.49	0.98	0.63	0.81	0.49	0.20	1.20	2.10	0.96
Southern Yellow Pine, 12.5% Concentration							Yellow Poplar, 5.0% Concentration					
Max.	1.0	1.0	1.0	5.0	2.0	2.0	6.5	21.0	0.5	21.0	0	9.8
Min.	0	0	0	2.0	0	0.4	2.5	16.0	0	0	0	3.7
$\bar{X}$	0.5	0.4	0.6	2.8	1.2	1.10	4.5	18.6	0.2	7.4	0	7.68
σ	0.32	0.49	0.49	1.17	0.75	0.64	1.50	1.84	0.25	7.66	0	2.81
Southern Yellow Pine, 5.6% Concentration Borax							Yellow Poplar, 12.5% Concentration					
Max.	33	1	2	3	4	8.6	1.0	0	1	3	0	1.0
Min.	0.5	0	1	1	1	0.7	0.5	0	0	1	0	0.3
$\bar{X}$	7.6	0.4	1.4	2.0	2.2	2.72	0.7	0	0.2	1.6	0	0.50
σ	12.71	0.49	0.49	0.89	1.17	3.15	0.24	0	0.40	0.08	0	0.14

Similar comparisons of reproducibility of the flame spread tests cannot be made since this test was performed by only one laboratory, but the values of the standard deviation and coefficient of variation for this series are shown in Table XXIII. The data for maximum temperature, which concern only the fire-tube test and "special" crib test, give average values of standard deviation for all tests conducted, as follows: fire tube test, $\sigma_x = 81$, $\bar{\sigma} = 70$; "special" crib test, $\sigma_x = 58$, $\bar{\sigma} = 37$.

The statistics given above serve to indicate the variations between tests and laboratories. Large variations resulted in the recorded times of duration of flaming and glowing in all tests for reasons discussed later. The variations in percentage loss of area in the timber test are similar to the variations in the percentage loss in weight.

TABLE IX.—FINAL WEIGHT LOSS IN SPECIAL CRIB TEST.

NOTE 1.—Values for final weight loss are given as percentages of the weight of the specimen.
NOTE 2.—For information on treatments of test boards, see Table I.
NOTE 3.—Absorption values are expressed as the weight in pounds of dry salt retained after treatment per cubic foot of wood.
NOTE 4.—Specific gravity is based on the air-dry weight (65 per cent relative humidity at 80 F.) and the air-dry volume.
NOTE 5.—The symbols used are as follows: $\bar{X}$ = average, σ = standard deviation, and v = coefficient of variation. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
SOUTHERN YELLOW PINE, UNTREATED							WHITE OAK, UNTREATED					
Board No.	52	39	48	56	46		23	5	22	24	12	
Sp. gr.	0.566	0.524	0.523	0.603	0.527	0.549	0.730	0.690	0.796	0.747	0.757	0.744
Moisture, per cent	6.9	6.2	8.4	7.65	7.3	7.3	8.9	7.3	7.4	7.1	8.0	7.7
Absorption	0	0	0	0	0	0	0	0	0	0	0	0
Weight loss, per cent:												
Max.	89	88.0	90.5	92	91	90.1	89.0	93.0	91.5	91.7	91.0	91.2
Min.	86.5	86.5	87.5	85.6	88	86.8	87.5	90.0	86.5	86.9	88.5	87.9
$\bar{X}$	87.4	87.3	88.9	89.4	89.2	88.44	88.1	91.4	89.9	88.9	89.9	89.64
σ	0.97	0.51	1.32	2.65	1.03	1.296	0.49	1.02	1.83	1.57	0.80	1.14
v	1.11	0.58	1.48	2.96	1.15	1.47	0.57	1.12	2.04	1.77	0.89	1.27
SOUTHERN YELLOW PINE, 2.5% CONCENTRATION							WHITE OAK, 12.5% CONCENTRATION					
Board No.	50	2	44	53	29		18	11	17	21	16	
Sp. gr.	0.573	0.590	0.591	0.570	0.585	0.582	0.688	0.800	0.720	0.645	0.719	0.714
Moisture, per cent	7.2	6.7	9.2	8.1	7.8	7.8	9.4	7.5	8.1	7.8	8.9	8.3
Absorption	1.065	1.06	1.075	1.08	1.065	1.069	1.395	1.52	2.05	1.49	1.70	1.631
Weight loss, per cent:												
Max.	78	74	77	76.3	74	75.9	87.0	86.0	89.5	89.6	86.0	87.6
Min.	72.5	73	75	73.4	73	73.4	85.0	81.0	68.5	83.9	82.5	80.2
$\bar{X}$	75.3	73.2	75.7	74.6	73.7	74.50	85.6	83.4	80.1	86.6	85.0	84.14
σ	1.91	0.40	0.87	0.98	0.40	0.912	0.80	1.85	7.37	1.87	1.30	2.64
v	2.54	0.55	1.15	1.31	0.54	1.22	0.93	2.22	9.23	2.16	1.53	3.14
SOUTHERN YELLOW PINE, 7.5% CONCENTRATION							YELLOW POPLAR, UNTREATED					
Board No.	26	10	19	28	15		21	3	7	25	6	
Sp. gr.	0.544	0.565	0.549	0.560	0.544	0.552	0.486	0.477	0.473	0.544	0.472	0.490
Moisture, per cent	8.8	7.0	8.9	6.8	9.3	8.2	6.2	5.7	6.1	6.0	8.6	6.5
Absorption	3.305	3.33	3.25	3.27	3.27	3.285	0	0	0	0	0	0
Weight loss, per cent:												
Max.	39.5	37.5	66.0	57.8	45.5	49.6	95.0	92.0	92.5	94.7	95.0	93.8
Min.	37.0	26.0	44.5	34.0	35.5	35.4	94.0	90.0	91.5	93.3	92.0	92.2
$\bar{X}$	38.54	31.2	61.0	41.92	39.6	42.45	94.3	91.4	91.9	94.1	94.0	93.14
σ	0.92	4.55	8.29	8.26	3.76	5.156	0.40	0.80	0.37	0.46	1.10	0.626
v	2.39	14.58	13.59	19.70	9.49	12.14	0.42	0.87	0.40	0.49	1.17	0.67
SOUTHERN YELLOW PINE, 12.5% CONCENTRATION							YELLOW POPLAR, 5.0% CONCENTRATION					
Board No.	32	9	30	36	13		16	4	14	17	9	
Sp. gr.	0.584	0.560	0.589	0.572	0.599	0.581	0.527	0.507	0.446	0.484	0.495	0.492
Moisture, per cent	9.4	6.9	8.4	8.3	9.9	8.6	7.8	6.2	6.6	8.4	10.1	7.8
Absorption	5.59	5.68	5.575	5.68	5.99	5.703	1.31	2.28	1.545	1.98	2.245	1.872
Weight loss, per cent:												
Max.	22.3	23	27	22.5	23.5	23.7	87.5	69.0	75	74.3	70	75.2
Min.	20.1	20	24	21	21	21.2	78.5	67.5	72	69.4	65	70.5
$\bar{X}$	21.3	21.4	25.4	21.7	22.2	22.41	82.7	68.1	73.0	72.1	68.6	72.90
σ	0.77	1.16	1.02	0.49	0.93	0.873	3.34	0.49	1.09	1.91	1.96	1.758
v	3.62	5.42	4.02	2.26	4.19	3.89	4.03	0.72	1.49	2.65	2.86	2.41
SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX							YELLOW POPLAR, 12.5% CONCENTRATION					
Board No.	35	1	31	47	20		23	8	13	24	10	
Sp. gr.	0.562	0.551	0.568	0.546	0.537	0.553	0.415	0.475	0.414	0.424	0.422	0.430
Moisture, per cent	10.8	9.5	9.5	10.1	10.2	10.0	10.8	6.2	7.1	7.7	10.8	8.5
Absorption	2.435	2.50	2.50	2.49	2.595	2.504	6.415	6.27	6.465	6.24	6.19	6.316
Weight loss, per cent:												
Max.	93	83.0	87.5	91.2	90	88.9	28	25	29.5	30.6	25	27.6
Min.	89	80.0	83	88.3	86	85.3	24.8	21	25	24.2	20	23.0
$\bar{X}$	90.6	81.8	85.7	90.3	87.8	87.24	26.7	23.1	26.9	27.1	22.0	25.16
σ	1.50	0.98	1.83	1.07	1.63	1.40	1.12	1.50	1.77	2.32	1.90	1.722
v	1.66	1.20	2.14	1.19	1.86	1.60	4.20	6.50	6.58	8.56	8.65	6.85

EFFECT OF TREATMENT

As previously stated, the treatments used in this investigation were intended to produce different degrees of resistance to fire exposure. The efficiency of the chemicals used, as compared with other treatments, is immaterial to the purposes of these tests, except that the fire-retardant effectiveness was intended to provide the means of comparing the practical application of the tests. The use of one type of treatment, the limited number of species and test samples, do not warrant the determination of functional relationships between the observations and degree of treatment. It may also be noted that all the different observations recorded from each test may not be required or desirable for a practical differentiation between treatments or the satisfaction of prescribed acceptance limits. The available information will, however, be useful in determining the more significant data.

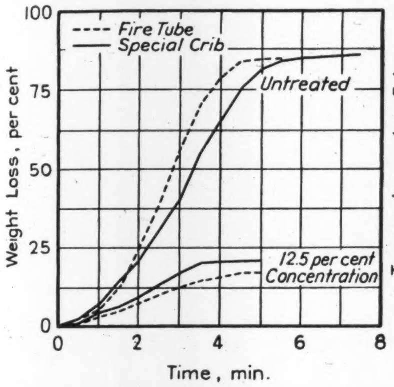

FIG. 10.—Typical Weight Loss and Temperature Curves for Fire Tube Tests and "Special" Crib Tests.

Material—Pine, untreated and treated with 12.5 per cent concentration of ammonium salts. Time of Removal of Burner: fire tube test, 4 min.; "special" crib test, 3 min.

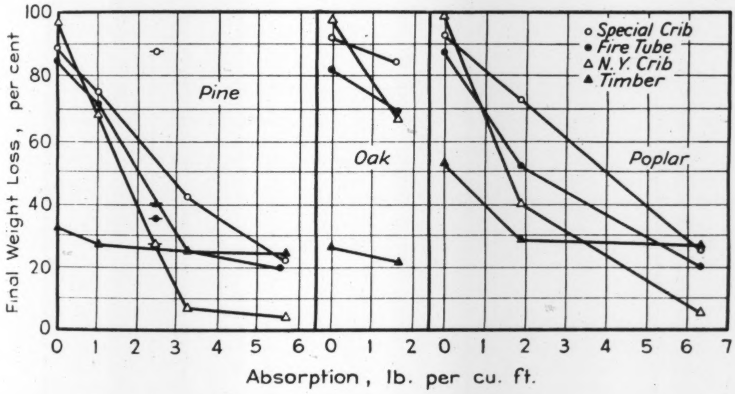

FIG. 11.—Final Weight Loss, per cent.

The relation between the various observations and the retention of chemical in each treatment is shown graphically in Figs. 10 to 20. These curves represent the average of the respective observations without correction for extreme values which, if made, however, would not appreciably alter the relative positions or the conclusions to be drawn. In the final weight loss *versus* treatment (Fig. 11) the relative positions of the curves for the four tests are essentially the same for the three species. A favorable spread of weight-loss values from untreated to well treated material results in the fire tube test, "special" crib test, New York crib test, and flame-spread test (see Fig. 20). The timber test shows very little differentiation between the various degrees of treatment. The range between maximum and minimum values recorded by individual laboratories or among laboratories is more than the difference between averages for all treatments. In the case of pine, the untreated material shows only slightly more loss than the material with best treatment. The curves for the fire-tube test, the New York crib test, and the flame-spread test tend to flatten over the higher concentrations of treatment, while the "special" crib test shows a spread of results at a rate nearly the same as for the less effective treatments. The smaller difference in weight loss per pound of retained chemical might be interpreted to indicate a loss in efficiency of retardants at higher concentration. While the efficiency per pound of salt would be expected to decrease as saturation is approached, the abrupt change in the weight loss beyond 3-lb. absorption

TABLE X.—DURATION OF FLAME IN SPECIAL CRIB TEST.

NOTE 1.—Values for duration of flame after removal of the burner are given in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table IX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average
SOUTHERN YELLOW PINE, UNTREATED						
Max	3.0	3.15	7.5	3.5	2.83	4.00
Min.	2.5	2.75	4.0	3.0	2.58	2.97
$\bar{X}$	2.63	2.93	6.20	3.26	2.75	3.55
σ	0.24	0.14	1.33	0.20	0.09	0.400
v	9.12	4.78	21.43	6.14	3.27	11.28
SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						
Max.	12.0	2.83	10.75	8.50	4.08	7.63
Min.	4.75	2.58	3.5	3.0	3.17	3.40
$\bar{X}$	9.70	2.71	8.05	5.32	3.78	5.91
σ	3.74	0.10	2.54	2.17	0.59	1.83
v	38.55	3.69	31.55	40.75	15.60	30.96
SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						
Max.	3.17	3.30	5.0	5.92	2.0	3.88
Min.	3.0	1.12	3.0	0	0	1.42
$\bar{X}$	3.03	2.14	4.35	2.98	0.40	2.58
σ	0.17	0.94	0.70	1.88	0.80	0.90
v	5.61	44.0	16.10	63.10	200.0	34.90
SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						
Max.	1.00	0.72	0.25	0.42	0.33	0.54
Min.	0.25	0.42	0.25	0	0	0.18
$\bar{X}$	0.52	0.58	0.25	0.214	0.07	0.33
σ	0.28	0.10	0	0.06	0.13	0.113
v	53.90	17.25	0	28.03	185.7	34.25
SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						
Max.	11.5	4.92	18.0	5.0	9.23	9.73
Min.	8.5	4.0	11.5	2.75	3.97	6.14
$\bar{X}$	9.70	4.48	13.20	3.57	6.23	7.44
σ	1.73	0.30	2.44	0.81	2.01	1.458
v	17.84	6.69	18.48	22.69	32.26	19.59

Laboratory	C	F	W	B	P	Average
WHITE OAK, UNTREATED						
Max	3.50	3.83	6.5	7.0	4.0	4.97
Min.	3.17	3.33	3.5	6.0	3.5	3.90
$\bar{X}$	3.33	3.60	4.20	6.30	3.82	4.25
σ	0.11	0.17	0.17	0.40	0.14	0.396
v	3.30	4.73	27.85	6.35	3.66	9.31
WHITE OAK, 12.5% CONCENTRATION						
Max.	16.0	5.58	27.12	13.50	21.17	16.67
Min.	14.0	4.50	12.00	3.59	10.97	9.01
$\bar{X}$	15.0	4.97	20.30	7.15	16.23	12.73
σ	0.63	0.43	5.45	4.24	3.70	2.889
v	4.20	8.65	26.85	59.30	22.80	22.67
YELLOW POPLAR, UNTREATED						
Max.	2.25	2.08	6.5	2.67	2.08	3.12
Min.	2.0	1.75	4.5	2.42	1.67	2.47
$\bar{X}$	2.08	1.92	5.8	2.57	1.83	2.84
σ	0.11	0.12	0.75	0.14	0.17	0.256
v	5.29	6.25	12.92	5.45	9.29	9.01
YELLOW POPLAR, 5.0% CONCENTRATION						
Max.	9.75	4.42	4.5	9.0	7.07	6.95
Min.	6.25	2.75	3.0	3.5	5.92	4.28
$\bar{X}$	8.62	3.29	3.90	5.88	6.46	5.63
σ	1.32	0.59	0.58	2.10	0.40	0.997
v	15.31	17.93	14.87	35.71	6.19	17.71
YELLOW POPLAR, 12.5% CONCENTRATION						
Max.	0.50	1.50	0	0.57	0.25	0.56
Min.	0.17	0.12	0	0	0	0.06
$\bar{X}$	0.28	0.68	0	0.31	0.14	0.35
σ	0.12	0.45	0	0.20	0.09	0.216
v	42.86	66.17	0	64.51	64.29	61.71

in the pine series and 2-lb. absorption in the poplar may be due to characteristics of the tests. In considering the poplar series, it should be noted that the intermediate treatment was omitted. The results for a similar intermediate treatment if determined would probably have resulted in relations similar to the pine series. In all of the tests, the total heat involved during the exposure of the sample includes the heat generated by the fuel composing the sample, as well as the exposure flame. For untreated or poorly treated material, the heat generated by the blazing sample is large and at its maximum rate while the burner is in place, whereas the burner flame is augmented to a lesser degree in the well treated material. This condition necessarily results in widely different exposures depending upon the behavior of the sample. The fire tube test, the New York crib test, and the flame spread test employ bunsen burner flames with less total heat content than the Meker burner flame used in the "special" crib test, and the variation of test conditions is therefore greater in these tests.

TABLE XI.—DURATION OF GLOW IN SPECIAL CRIB TEST.

NOTE 1.—Values for duration of glow after flaming ceased are given in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table IX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average
SOUTHERN YELLOW PINE, UNTREATED						
Max.	26.5	14.35	31.5	16.0	21.38	21.95
Min.	13.5	8.33	16.5	13.75	15.58	13.53
$\bar{X}$	20.57	11.61	22.40	15.23	17.44	17.45
σ	5.13	1.94	5.02	1.10	2.06	3.25
v	24.94	16.71	22.41	7.22	11.81	18.62
SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						
Max.	2.5	11.92	1.0	2.33	3.97	4.34
Min.	0	1.08	0	0.50	0.42	0.40
$\bar{X}$	0.95	5.94	0.20	1.25	1.95	2.06
σ	1.08	4.07	0.40	0.63	1.24	1.485
v	113.68	68.52	200.0	50.40	63.59	72.09
SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						
Max.	0.17	0	1.5	0	3.2	0.97
Min.	0	0	0	0	0.57	0.11
$\bar{X}$	0.03	0	0.50	0	1.92	0.49
σ	0.07	0	0.63	0	0.86	0.311
v	233.3	0	126.0	0	44.79	63.47
SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						
Max.	0.08	0	0	3.42	0.68	0.84
Min.	0	0	0	0	0.23	0.05
$\bar{X}$	0.05	0	0	3.28	0.52	0.77
σ	0.04	0	0	0.14	0.17	0.070
v	80.0	0	0	4.27	32.69	9.09
SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						
Max.	59.0	40.0	54.5	44.0	52.6	5.00
Min.	35.5	18.0	33.0	37.3	43.9	33.5
$\bar{X}$	45.70	23.60	42.50	40.33	47.82	39.99
σ	8.37	8.24	7.94	2.15	2.89	5.92
v	18.32	34.92	18.68	5.33	6.04	14.80

Laboratory	C	F	W	B	P	Average
WHITE OAK, UNTREATED						
Max.	18.67	17.0	32.0	20.0	35.4	24.61
Min.	14.5	11.0	17.5	15.0	30.3	17.7
$\bar{X}$	17.27	12.80	23.50	17.30	31.62	20.50
σ	1.82	2.23	5.54	1.78	1.92	2.66
v	10.54	17.42	23.57	10.29	6.07	12.98
WHITE OAK, 12.5% CONCENTRATION						
Max.	13.0	20.0	22.0	23.4	21.1	19.9
Min.	7.0	14.0	2.0	9.0	11.6	8.7
$\bar{X}$	10.0	18.4	11.5	17.2	14.6	14.33
σ	2.19	2.24	6.54	5.45	3.40	3.96
v	21.9	12.17	56.87	31.69	23.28	27.63
YELLOW POPLAR, UNTREATED						
Max.	22.83	12.50	7.5	23.42	19.08	17.07
Min.	11.0	5.12	5.5	16.58	14.23	10.49
$\bar{X}$	16.12	8.11	6.70	19.93	16.82	13.54
σ	4.53	2.69	0.75	2.43	1.85	2.449
v	28.10	33.17	11.19	12.19	11.00	18.09
YELLOW POPLAR, 5.0% CONCENTRATION						
Max.	14.25	6.91	7.5	....	3.77	8.11
Min.	6.75	0.86	6.0	....	1.40	3.75
$\bar{X}$	10.48	4.46	6.70	....	2.50	6.04
σ	2.39	2.00	0.51	....	0.75	1.412
v	22.81	44.84	7.61	....	30.0	23.37
YELLOW POPLAR, 12.5% CONCENTRATION						
Max.	0.25	0	0	0	0.12	0.07
Min.	0	0	0	0	0	0
$\bar{X}$	0.07	0	0	0	0.02	0.02
σ	0.10	0	0	0	0.12	0.044
v	142.86	0	0	0	600.0	220.0

The higher weight loss per pound of salt and also the higher rate of weight loss for the lower salt concentrations in the case of the fire tube may also be due to the "flue" effect which is accentuated with higher temperatures. Laboratory C noted a decided blast effect in the untreated pine series, resulting in a momentary rise of temperature at the top of the tube of from 80 to 140 F. when the

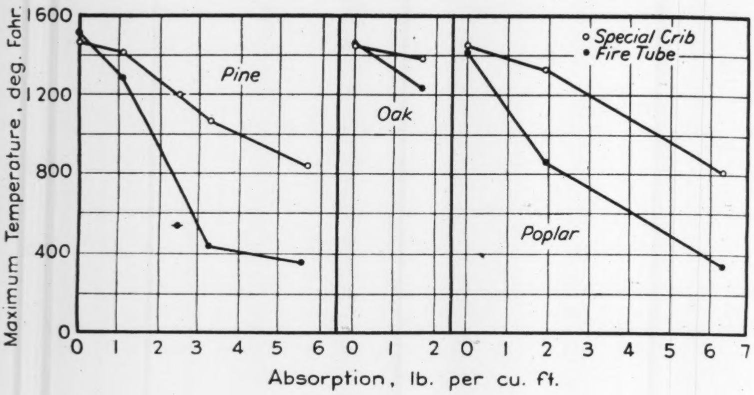

FIG. 12.—Maximum Temperature.

TABLE XII.—MAXIMUM TEMPERATURE IN SPECIAL CRIB TEST.

NOTE 1.—Values for maximum temperatures are given in degrees Fahrenheit.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table IX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
SOUMHERN YELLOW PINE, UNTREATED							WHITE OAK, UNTREATED					
Max.	1485	1490	1472	1480	1490	1483.4	1535	1475	1472	1493	1460	1487.0
Min.	1450	1450	1445	1462	1475	1456.4	1490	1385	1238	1445	1420	1395.6
$\bar{X}$	1468	1472	1459	1472	1481	1470.4	1511	1443	1409	1466	1450	1456.0
σ	12.08	14.40	10.80	6.43	4.90	9.72	16.56	30.80	87.80	18.87	15.49	33.90
v	0.82	0.98	0.74	0.44	0.33	0.66	1.10	2.13	6.23	1.29	1.08	2.33
SOUTHERN YELLOW PINE, 2.5% CONCENTRATION							WHITE OAK, 12.5% CONCENTRATION					
Max.	1455	1465	1373	1440	1450	1436.6	1450	1465	1382	1445	1410	1430.4
Min.	1445	1425	1292	1393	1380	1387.0	1400	1385	1112	1350	1330	1315.4
$\bar{X}$	1451	1443	1333	1412	1424	1412.6	1431	1434	1281	1402	1379	1385.4
σ	3.74	14.70	30.60	17.46	24.17	18.13	18.55	28.50	97.0	30.66	28.00	40.54
v	0.26	1.02	2.30	1.24	1.70	1.28	1.30	1.99	7.57	2.19	2.03	5.93
SOUTHERN YELLOW PINE, 7.5% CONCENTRATION							YELLOW POPLAR, UNTREATED					
Max.	1080	1175	1148	1114	1100	1123.4	1510	1380	1454	1524	1485	1470.6
Min.	950	1100	1040	1013	950	1010.6	1475	1345	1364	1502	1460	1429.2
$\bar{X}$	1011	1136	1108	1062	1022	1067.8	1497	1357	1422	1511	1471	1451.6
σ	41.76	29.60	43.0	33.86	52.69	40.18	11.7	12.1	34.9	7.4	10.2	15.26
v	4.11	2.61	3.88	3.19	5.16	3.76	0.78	0.89	2.45	0.49	0.69	1.05
SOUTHERN YELLOW PINE, 12.5% CONCENTRATION							YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	890	1060	986	725	870	906.2	1485	1330	1364	1332	1340	1370.2
Min.	815	785	806	682	660	749.6	1420	1155	1256	1242	1220	1258.6
$\bar{X}$	854	952	894	706	798	840.8	1458	1257	1332	1287	1286	1324.0
σ	30.89	98.70	70.70	13.94	72.50	57.35	23.79	61.50	44.70	34.24	39.80	40.81
v	3.62	10.37	7.91	1.97	9.09	6.82	1.63	4.89	3.36	2.66	3.02	3.08
SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX							YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	1370	1350	1148	1307	1210	1277.0	905	1040	806	840	760	870.2
Min.	1280	1195	932	1017	1040	1092.8	815	855	716	682	680	749.6
$\bar{X}$	1336	1254	1076	1224	1107	1199.4	863	976	756	741	720	811.2
σ	31.53	60.90	82.10	104.98	60.63	68.03	28.74	64.60	31.0	58.25	31.62	42.84
v	2.36	4.86	7.63	8.58	5.48	5.67	3.33	6.62	4.10	7.86	4.39	5.28

sample was practically consumed, indicating the presence of unburned gases in the tube. A wide spread of results between poorly treated and well treated material, whatever the cause, is desirable, but the abrupt flattening of the curves is more likely to be due to change in test conditions brought about by the sample itself than to decided decrease in efficiency of the treatment. The timber test on the other hand represents the other extreme in relative heat generated by the sample and the test flame. While the furnace temperature raises slightly during the test due to the combustion of the sample, the attack by the furnace flame is more severe than in any of the other tests and the heat from the sample is relatively small. The exposure conditions are therefore more uniform but apparently afford little differentiation between treatments. It may be noted also that the final weight loss for untreated material was consistently highest for the New York crib test. The more complete consumption of material in this test is probably due to the form of sample and free access of air. The borax-treated pine samples do not behave according to the variation of ammonium sulfate - monoammonium phosphate treatment as indicated by the plotted points, the borax treatment being generally less effective due to the tendency for continued glowing.

TABLE XIII.—AREA UNDER TIME-TEMPERATURE CURVE IN SPECIAL CRIB TEST.

NOTE 1.—Values for area under the time-temperature curve include only that portion above the 600 F. line, and are expressed as the product of the temperature in degrees Fahrenheit multiplied by the time in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table IX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	3410	3960	3630	4183	3810	3798.6	4480	4412	4342	4631	4825	4538.0
Min.	3280	3720	3344	3897	3310	3510.2	3350	3924	2995	4395	3910	3714.8
$\bar{X}$	3362	3860	3494	4039	3622	3675.4	4206	4256	3935	4504	4239	4228.0
σ	527	84.8	84.6	103.8	187.3	102.64	430.0	174.8	489.6	87.9	335.2	303.50
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	3200	3568	2776	4656	3180	3476.0	4180	4628	4194	3834	3590	4085.2
Min.	2850	3332	2520	3075	2680	2891.4	3680	3980	745	3274	3325	3000.8
$\bar{X}$	3090	3420	2671	3441	2994	3123.2	3912	4420	3020	3573	3485	3682.0
σ	131.9	79.2	105.4	609.1	195.2	224.16	162.0	236.0	1242.0	221.2	90.4	390.32
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	710	868	1202	685	725	838.0	3700	3128	3211	3984	3260	3456.6
Min.	500	640	680	448	500	553.6	3180	2732	2761	3635	2910	3043.6
$\bar{X}$	588	732	1049	553	623	709.0	3366	2952	3033	3819	3088	3251.6
σ	96.2	78.4	187.0	94.1	76.7	106.48	176.0	150.8	162.0	127.9	151.6	153.66
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	400	704	500	149	260	402.6	3690	2596	3071	2801	2445	2920.6
Min.	210	156	256	50	55	145.4	3150	1780	2272	1880	1460	2108.4
$\bar{X}$	326	424	361	99	194	280.8	3398	2288	2722	2398	2183	2597.8
σ	71.7	178.0	86.4	34.9	72.5	88.7	173.0	307.6	283.1	375.0	366.4	301.02
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	2850	2688	1958	5724	1940	3032.0	450	880	230	274	265	419.8
Min.	2150	2232	479	2016	635	1502.4	300	352	83	13	90	167.6
$\bar{X}$	2598	2404	1399	2994	1186	2116.2	388	660	176	164	153	308.2
σ	251.9	154.8	517.9	1376.4	522.7	564.74	51.0	177.6	52.19	98.4	71.3	90.24

The maximum temperature and maximum rate of weight loss, determined in the fire-tube test and "special" crib test, differentiate between treatments as shown in Figs. 12 and 13. The "special"

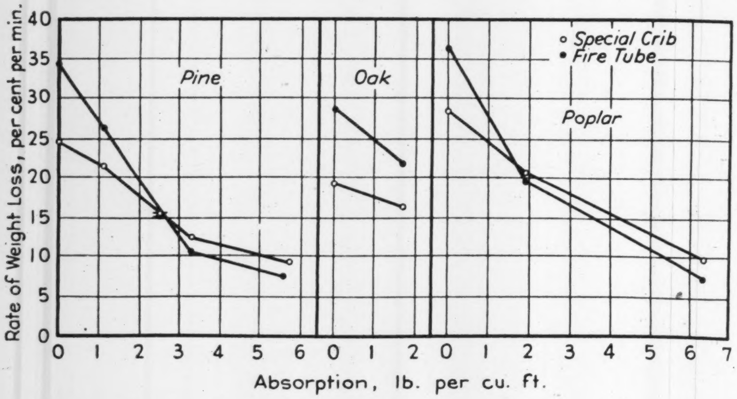

FIG. 13.—Maximum Rate of Weight Loss.

TABLE XIV.—MAXIMUM RATE OF WEIGHT LOSS IN SPECIAL CRIB TEST.

NOTE 1.—Values for maximum rate of weight loss are expressed as the loss of weight per minute, calculated as a percentage of the original weight of the specimen.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table IX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
SOUTHERN YELLOW PINE, UNTREATED							WHITE OAK, UNTREATED					
Max.	29.0	28.0	28.0	26.0	26.0	27.4	20.0	20.0	22.0	20.0	19.0	20.2
Min.	24.0	25.0	21.0	20.0	23.0	22.6	19.0	17.0	18.0	18.0	17.0	17.8
$\bar{X}$	26.6	26.8	23.8	22.4	24.6	24.84	19.8	18.4	20.0	19.6	18.0	19.16
σ	1.74	1.17	2.48	2.53	1.04	1.79	0.40	1.02	1.40	0.80	0.63	0.85
SOUTHERN YELLOW PINE, 2.5% CONCENTRATION							WHITE OAK, 12.5% CONCENTRATION					
Max.	24.0	22.0	24.0	22.0	25.0	23.4	19.0	16.0	15.0	20.0	20.0	18.0
Min.	20.0	20.0	19.0	19.0	20.0	19.6	16.0	14.0	10.0	19.0	16.0	15.0
$\bar{X}$	21.8	21.2	21.4	20.6	22.4	21.49	17.2	15.2	12.8	19.8	17.2	16.44
σ	1.33	0.98	1.85	1.20	3.04	1.68	1.85	0.75	2.08	0.40	2.16	1.45
SOUTHERN YELLOW PINE, 7.5% CONCENTRATION							YELLOW POPLAR, UNTREATED					
Max.	11.0	12.0	16.0	14.0	17.0	13.8	29.0	32.0	31.0	32.0	30.0	30.8
Min.	11.0	8.0	13.0	10.0	12.0	10.8	28.0	28.0	28.0	22.0	28.0	26.8
$\bar{X}$	11.0	10.8	14.0	12.2	14.4	12.48	28.2	29.6	29.4	26.0	29.0	28.44
σ	0	1.60	1.26	1.30	2.64	1.36	0.40	1.50	1.02	4.20	0.89	1.602
SOUTHERN YELLOW PINE, 12.5% CONCENTRATION							YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	9.0	12.0	12.0	8.0	12.0	10.6	24.0	22.0	24.0	26.0	24.0	24.0
Min.	8.0	7.0	10.0	8.0	9.0	8.4	21.0	16.0	20.0	14.0	18.0	17.8
$\bar{X}$	8.24	8.80	10.40	8.00	10.40	9.17	22.6	20	22.6	18.4	19.6	20.64
σ	0.39	1.72	0.80	0	1.04	0.79	1.36	2.53	1.50	4.27	2.25	2.381
SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX							YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	20.0	18.0	14.0	22.0	22.0	19.2	11.0	10.0	12.0	14.0	10.0	11.4
Min.	15.0	12.0	11.0	12.0	12.0	12.4	9.0	8.0	9.0	8.0	9.0	8.6
$\bar{X}$	16.8	14.8	12.8	17.2	14.4	15.20	9.8	8.8	9.8	10.6	9.6	9.72
σ	1.78	2.04	1.17	3.50	3.88	2.47	0.75	0.75	1.17	1.96	0.49	1.024

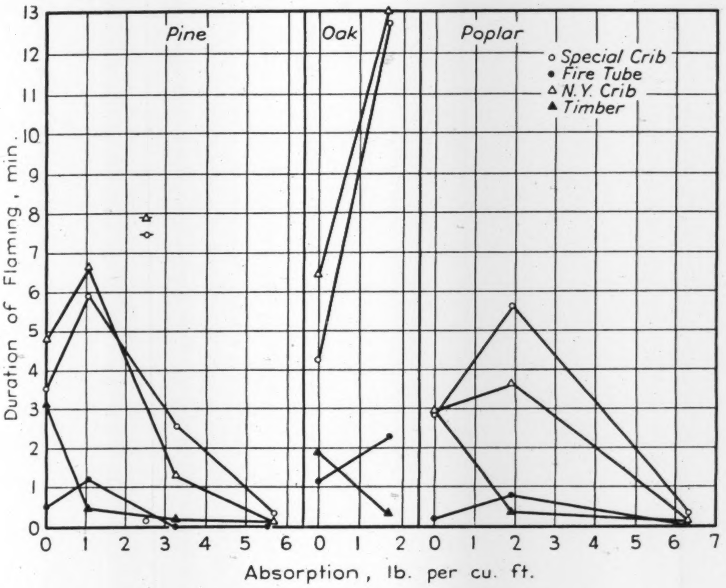

FIG. 14.—Duration of Flaming.

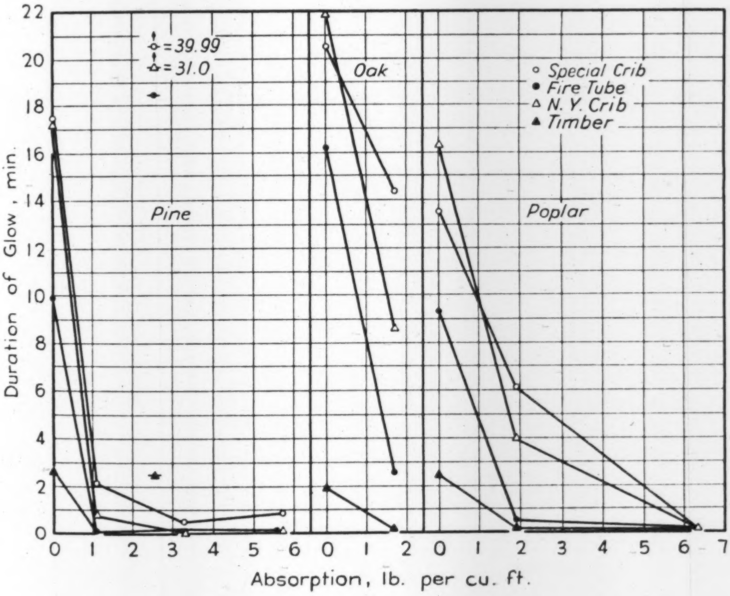

FIG. 15.—Duration of Glowing.

crib test gives more nearly a linear relationship as in the final weight loss curves, although with somewhat less spread of results, due perhaps to the "flue" action of the fire tube in testing poorly treated material. The rate of weight loss in the flame-spread test is similar to the others for the pine and oak, although with less spread of results, and irregular in the case of the poplar.

TABLE XV.—WEIGHT LOSS AFTER REMOVAL OF BURNER IN SPECIAL CRIB TEST.

NOTE 1.—Values for weight loss after removal of the burner are expressed as percentages of the original weights of the specimens.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table IX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	49.0	50.0	54.5	58.6	50.0	52.4	56.0	61.0	64.5	60.7	62.5	60.9
Min.	44.5	49.0	50.0	53.2	43.5	48.0	50.2	58.0	55.0	53.5	58.5	55.0
$\bar{X}$	46.3	49.7	51.9	55.5	47.5	50.16	54.44	59.8	61.6	56.5	61.0	58.67
σ	1.69	0.51	1.53	1.96	2.21	1.581	2.21	1.17	3.51	2.89	1.38	2.232
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	37.5	41.0	41.5	40.0	36.0	39.2	60.5	64.0	65.5	57.6	59.0	61.3
Min.	34.0	38.0	38.5	33.4	32.0	35.2	52.5	62.0	47.5	51.4	53.5	53.4
$\bar{X}$	35.9	38.8	40.0	37.0	33.0	36.94	55.54	63.0	57.70	55.34	56.40	57.60
σ	1.17	1.17	1.14	2.54	1.52	1.507	2.97	0.71	6.13	2.11	2.27	2.837
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	16.0	14.5	34.5	33.8	18.5	23.5	39.0	44.0	42.0	48.2	38.0	42.2
Min.	15.0	6.0	15.5	13.4	8.0	11.6	35.0	36.0	35.5	40.3	29.5	35.3
$\bar{X}$	15.34	9.50	30.2	18.8	13.0	17.37	37.9	40.0	38.8	45.6	34.7	39.40
σ	0.42	4.23	7.40	7.67	3.65	4.673	1.50	3.57	2.60	2.87	2.89	2.685
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	4.5	4.0	3.0	3.5	3.5	3.7	46.5	42.0	40.0	39.1	35.0	40.75
Min.	3.3	2.0	1.0	1.8	2.0	2.0	39.5	30.5	30.0	28.9	31.0	32.0
$\bar{X}$	3.84	3.00	2.20	2.84	2.70	2.92	43.2	37.1	35.5	35.1	33.1	36.80
σ	0.39	0.63	0.68	0.57	0.60	0.574	2.68	3.98	3.33	3.37	1.80	3.032
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	67.0	62.0	63.0	63.8	64.0	64.0	5.0	6.5	6.0	8.5	2.5	5.7
Min.	61.8	58.5	57.0	60.0	61.0	59.7	3.3	1.5	4.0	3.1	0	2.4
$\bar{X}$	64.8	60.0	61.2	61.8	62.7	62.09	4.1	2.9	4.9	4.6	0.7	3.44
σ	2.00	1.64	2.22	1.60	1.08	1.707	0.60	1.82	0.92	2.10	0.98	1.284

The duration of flaming and glowing after removal of the burner flame, or removal from the furnace in the timber test, are generally less consistent than the other measurements (see Figs. 14 and 15). This is due to more or less favorable conditions in the respective tests and to difficulties in making these observations. Some observers considered the short blue flame which usually follows the blazing yellow flame as being associated with glowing, while others included both kinds of flame in the time of flaming. In the timing of glow, some of the poorly treated samples continued to glow for long periods, and consequently some of the results may not represent the entire duration. The conditions afforded by the two types of crib test appear to be more favorable to continued flaming and glowing than the fire tube test and timber test. In addition to the difficulty of observing flaming and glowing of the remainder of the sample hanging within the fire tube, this test shows little differentiation in treatment. The duration of flaming and glowing of treated material by this test may be affected by the presence of evolved gases within the tube that are

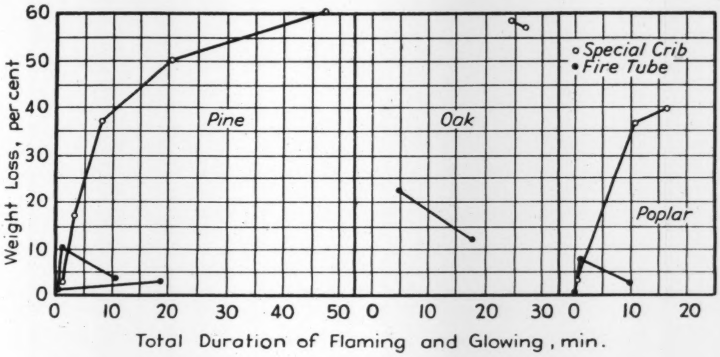

FIG. 16.—Weight Loss after Removal of Burner *versus* Time of Flaming plus Glowing.

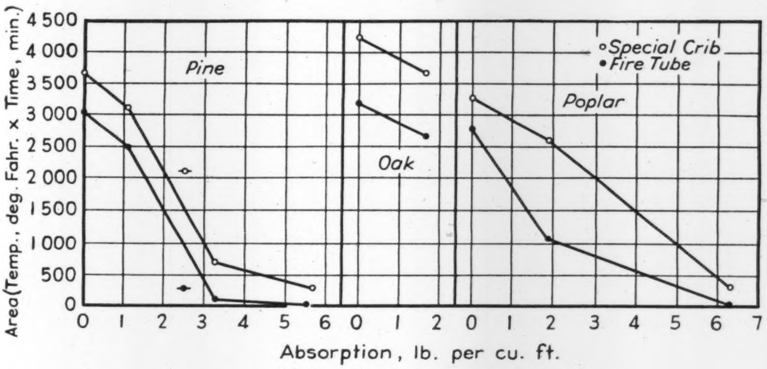

FIG. 17.—Area Under Time-Temperature Curve.

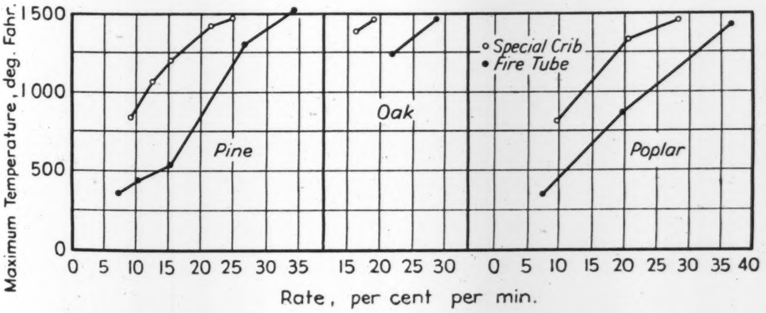

FIG. 18.—Rate of Weight Loss *versus* Maximum Temperature.

not expelled by the reduced draft action. The spread of results is likewise not great for the timber test, although somewhat better than indicated by the final weight loss. The influence of continued glowing on the final weight loss recorded by the "special" crib test has been noted.

TABLE XVI.—FINAL WEIGHT LOSS IN NEW YORK CRIB TEST.

NOTE 1.—Values for final weight loss are given as percentages of the weight of the specimen.

NOTE 2.—For the specimens used in the New York crib test, the board numbers, specific gravity values, and absorption of treating salt are the same as for the specimens used in the "special" crib test (see Table IX).

NOTE 3.—The symbols used are as follows: $\bar{X}$ = average, σ = standard deviation, and v = coefficient of variation. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average
SOUTHERN YELLOW PINE, UNTREATED						
Moisture, per cent	7.3	6.2	8.1	6.0	6.97	6.91
Weight loss, per cent:						
Max.	96.3	97.4	99.0	97.9	98.2	97.76
Min.	92.5	95.0	98.0	96.1	96.5	95.62
$\bar{X}$	94.82	96.20	98.44	97.10	97.22	96.76
σ	1.42	0.90	0.40	0.80	0.66	0.835
v	1.50	0.94	0.41	0.82	0.68	0.854
SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						
Moisture, per cent	7.8	6.7	8.8	6.4	6.95	7.33
Weight loss, per cent:						
Max.	65.8	73.2	76.1	71.8	67.5	70.88
Min.	58.7	63.6	70.1	65.9	60.0	63.66
$\bar{X}$	62.36	70.40	73.58	70.10	64.04	68.09
σ	2.75	3.54	2.24	2.16	2.42	2.621
v	4.41	5.03	3.05	3.08	3.78	3.850
SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						
Moisture, per cent	9.5	7.0	8.5	4.9	6.7	7.32
Weight loss, per cent:						
Max.	9.7	6.76	11.0	11.2	5.97	8.93
Min.	4.7	4.87	8.3	8.5	4.4	6.15
$\bar{X}$	6.3	5.53	9.14	10.10	4.81	7.18
σ	1.91	0.67	0.97	1.01	0.56	1.025
v	30.65	12.12	10.61	10.00	11.63	14.28
SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						
Moisture, per cent	9.0	6.9	8.3	6.9	9.7	8.16
Weight loss, per cent:						
Max.	2.6	4.0	5.4	5.2	4.46	4.33
Min.	2.1	3.0	3.8	4.5	3.0	3.28
$\bar{X}$	2.38	3.50	4.58	4.90	3.68	3.81
σ	0.17	0.35	0.57	0.50	0.57	0.431
v	7.14	10.00	12.42	10.02	15.49	11.30
SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						
Moisture, per cent	8.7	9.5	8.0	9.4	8.7	8.86
Weight loss, per cent:						
Max.	24.0	85.8	80.0	73.2	65.5	65.70
Min.	5.5	12.0	18.6	20.3	8.6	13.00
$\bar{X}$	11.78	43.20	61.16	42.90	43.16	40.44
σ	6.73	26.66	22.94	20.04	22.50	19.775
v	57.10	61.75	37.50	46.80	52.20	48.90

Laboratory	C	F	W	B	P	Average
WHITE OAK, UNTREATED						
Moisture, per cent	7.3	7.3	7.4	6.33	7.5	7.17
Weight loss, per cent:						
Max.	98.8	98.1	98.5	97.9	98.3	98.32
Min.	98.1	97.3	98.5	97.3	97.5	97.74
$\bar{X}$	98.42	97.80	98.50	97.56	97.92	98.04
σ	0.27	0.34	0	0.25	0.30	0.232
v	0.27	0.35	0	0.26	0.31	0.237
WHITE OAK, 12.5% CONCENTRATION						
Moisture, per cent	6.3	7.5	7.9	6.6	6.73	7.21
Weight loss, per cent:						
Max.	78.1	82.8	71.5	86.4	81.4	80.04
Min.	62.5	68.2	7.9	31.6	70.8	48.20
$\bar{X}$	71.28	75.60	43.76	69.00	75.88	67.10
σ	5.16	4.81	29.05	19.99	3.39	12.48
v	7.24	6.36	6.64	2.90	4.47	18.60
YELLOW POPLAR, UNTREATED						
Moisture, per cent	5.3	5.7	7.1	5.6	6.65	6.07
Weight loss, per cent:						
Max.	99.4	98.9	99.9	99.5	99.6	99.46
Min.	98.9	98.6	99.3	99.3	99.2	99.06
$\bar{X}$	99.12	98.80	99.62	99.38	99.44	99.27
σ	0.20	0.15	0.22	0.10	0.17	0.169
v	0.20	0.15	0.22	0.10	0.17	0.170
YELLOW POPLAR, 5.0% CONCENTRATION						
Moisture, per cent	5.8	6.2	6.8	6.6	9.5	6.98
Weight loss, per cent:						
Max.	86.9	23.2	84.5	36.4	27.4	51.68
Min.	75.9	12.5	34.1	12.9	14.0	29.88
$\bar{X}$	82.76	16.18	61.58	20.20	22.04	40.55
σ	3.63	3.82	16.42	8.49	5.05	7.480
v	4.39	23.60	26.65	42.00	22.90	18.43
YELLOW POPLAR, 12.5% CONCENTRATION						
Moisture, per cent	5.5	6.20	8.2	6.4	9.55	7.17
Weight loss, per cent:						
Max.	3.8	6.42	6.8	5.3	7.7	6.00
Min.	2.6	4.92	5.4	3.6	7.0	4.70
$\bar{X}$	3.04	5.51	5.92	4.50	7.44	5.28
σ	0.48	0.50	0.47	0.86	0.25	1.403
v	15.80	9.07	7.94	19.11	3.36	26.60

The flaming and glowing of the samples after removal of the burner is a visual indication of continued combustion at different rates. While these two rates of combustion have usually been differentiated, particularly in respect to flame duration or spread, the difficulties of measurement have been reported by most of the operators. Since continued combustion results in a continued loss in weight of the sample, such loss might be used as an index of the tendency of the sample to support flame and glow after removal of the ignition flame. Figure 16 shows the relation between the weight loss after removal of the burner and the total combined time of flaming and glowing recorded during the fire tube test and "special" crib test. The results of the fire tube test are inconsistent (points not in order of treatment). The "special" crib test results give a regular relationship, which for well-treated material can be taken as linear. In this test the weight loss after burner removal could be used as an index of continued support of combustion.

TABLE XVII.—DURATION OF FLAME IN NEW YORK CRIB TEST.

NOTE 1.—Values for the duration of flame after removal of the burner are given in minutes.
NOTE 2.—The data in this table were obtained from the specimens referred to in Table XVI. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	7.0	5.0	5.5	6.92	4.62	5.81	8.5	7.42	7.5	7.08	7.37	7.57
Min.	5.50	4.08	3.5	3.30	3.67	4.01	6.17	4.98	7.25	4.63	5.45	5.69
$\bar{X}$	6.35	4.55	4.15	4.88	4.14	4.81	6.93	5.83	7.35	5.59	6.40	6.42
σ	0.54	0.33	0.83	1.21	0.42	0.665	0.83	0.92	0.12	0.81	0.64	0.665
v	8.50	7.25	20.00	24.80	10.12	13.82	11.97	15.79	1.63	14.23	10.00	10.33
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	7.5	9.08	7.33	8.58	9.67	8.43	18.0	18.58	20.25	13.30	13.87	16.80
Min.	5.75	4.00	5.67	5.13	5.72	5.25	14.0	10.50	2.25	10.23	7.77	8.95
$\bar{X}$	6.90	6.03	6.45	6.47	7.30	6.63	15.30	13.45	11.60	12.10	12.59	13.01
σ	0.68	1.76	0.70	1.20	1.34	1.136	1.66	3.24	6.89	0.98	2.44	3.041
v	9.85	29.20	10.85	18.58	18.35	17.15	10.84	24.10	59.30	8.10	19.40	23.30
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	2.08	1.17	2.67	2.58	1.15	1.93	3.5	4.75	2.75	3.00	2.90	3.58
Min.	0.05	0.32	1.00	1.62	0.58	0.71	2.5	2.17	2.50	2.67	2.13	2.39
$\bar{X}$	1.28	0.69	1.52	2.04	0.89	1.28	3.10	3.11	2.65	2.84	2.47	2.83
σ	0.68	0.38	0.61	0.40	0.20	0.454	0.41	0.87	0.12	0.12	0.27	0.357
v	53.10	55.10	40.10	19.60	22.45	35.45	13.21	27.95	4.53	4.23	10.92	12.60
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	0	0.22	0	0.25	0.08	0.11	8.0	2.82	5.83	3.82	3.57	4.61
Min.	0	0.02	0	0.06	0	0.02	5.25	2.17	2.0	2.12	1.68	2.64
$\bar{X}$	0	0.11	0	0.15	0.02	0.06	6.70	2.39	3.48	2.99	2.68	3.65
σ	0	0.074	0	0.066	0.032	0.034	0.97	0.24	1.32	0.63	0.44	0.721
v	0	67.20	0	44.0	160.0	56.67	14.48	10.03	37.95	21.05	16.42	19.72
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	6.25	13.17	21.75	11.93	18.92	14.40	0	0.32	0.08	0.17	0	0.11
Min.	1.08	1.48	2.75	2.46	1.50	1.85	0	0	0	0	0	0
$\bar{X}$	2.73	6.67	14.20	7.69	8.05	7.87	0	0.23	0.02	0.09	0	0.07
σ	1.92	3.82	6.49	3.51	6.32	4.412	0	0.127	0.03	0.071	0	0.046
v	70.40	57.20	45.70	45.70	78.50	56.10	0	55.30	150.0	78.9	0	65.70

The areas under the time-temperature curves for the fire tube and "special" crib tests show relationships to treatment similar to final weight loss (see Fig. 17). The area resulting from the fire tube test on the 12.5 per cent concentration treatment of the pine and poplar was indeterminate since the maximum temperature was less than the starting temperature of 180 C. at the top of the tube, indicating an absorption of heat from the burner flame by the specimen not offset by heat generated by the specimen.

The relation between maximum temperature and rate of weight loss is shown in Fig. 18. While an approach to a correlation is indicated which might make it possible to use weight loss measurements to serve as an index of maximum temperature as in the case of duration of flaming and glowing, the elimination of temperature observations would probably be undesirable in both the fire tube and the "special" crib tests because of the means these measurements afford in controlling the burner flame. Such measurements, however, might only be required before and after test for such control if temperatures during the test were not considered necessary.

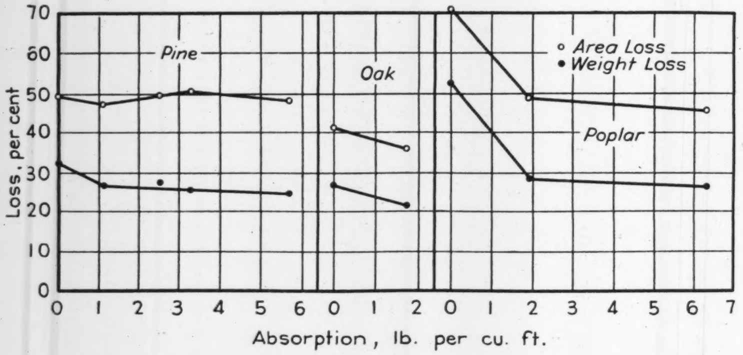

FIG. 19.—Timber Tests: Loss of Weight and Loss of Area.

TABLE XVIII.—DURATION OF GLOW IN NEW YORK CRIB TEST.

NOTE 1.—Values for duration of glow after flaming ceased are given in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table XVI. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	13.0	19.28	25.5	25.11	24.10	21.40	27.50	24.65	22.75	21.53	28.60	25.00
Min.	9.0	12.08	15.5	16.0	18.42	14.20	21.0	17.30	16.50	18.20	22.23	19.04
$\bar{X}$	10.77	16.38	19.20	19.30	20.49	17.23	23.07	21.72	19.40	19.34	25.63	21.83
σ	1.45	2.60	3.37	3.16	2.06	2.528	2.29	2.45	1.43	1.11	2.33	1.921
v	13.47	15.90	17.55	16.38	10.02	14.67	9.93	11.28	7.38	5.74	9.09	8.79
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	0.33	4.0	0.25	2.58	3.03	2.04	10.0	25.08	5.0	15.13	14.53	13.94
Min.	0.17	0.75	0	0	0	0.18	5.0	6.67	0	5.7	6.67	4.80
$\bar{X}$	0.20	2.03	0.05	0.72	0.94	0.79	7.20	13.55	2.25	10.43	9.51	8.59
σ	0.07	1.54	0.10	0.93	1.08	0.744	1.81	6.47	1.96	3.05	2.63	3.184
v	35.0	75.9	200.0	129.2	115.0	94.10	25.15	47.70	87.1	29.20	27.6	36.70
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	0	0.03	0	0	0	0.01	17.0	18.32	15.25	22.0	21.87	18.88
Min.	0	0	0	0	0	0	14.50	14.58	11.5	17.92	12.13	14.12
$\bar{X}$	0	0.02	0	0	0	0.004	15.55	15.99	13.75	19.98	16.56	16.37
σ	0	0.014	0	0	0	0.003	0.84	1.29	1.35	1.78	3.41	1.735
v	0	70.0	0	0	0	75.0	5.40	8.08	9.82	8.91	20.60	10.60
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Max.	0.05	0.15	0	0.03	0.33	0.11	11.0	0.50	8.5	0	0.35	4.07
Min.	0	0	0	0	0	0	6.75	0	4.25	0	0	2.20
$\bar{X}$	0.016	0.040	0	0.020	0.120	0.039	9.25	0.10	6.02	0	0.17	3.89
σ	0.020	0.056	0	0.010	0.112	0.040	1.94	0.20	1.41	0	0.14	0.922
v	125.0	140.0	0	50.0	93.4	102.5	21.0	200.0	23.40	0	82.5	23.70
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	41.25	38.30	43.5	41.97	44.55	41.91	0.03	0.02	0	0	0.15	0.04
Min.	6.67	23.60	30.5	26.23	19.83	21.36	0	0	0	0	0.08	0.02
$\bar{X}$	24.32	29.61	33.90	33.71	33.47	31.00	0.016	0.010	0	0	0.110	0.027
σ	14.00	5.43	4.88	5.13	7.98	5.483	0.013	0.010	0	0	0.026	0.010
v	57.6	18.3	14.4	15.2	23.8	17.68	81.3	100.0	0	0	23.6	37.05

Figure 19 shows the variation of weight loss and also loss of cross-sectional area for the different treatments as determined by the timber test. Both curves are essentially similar, with somewhat more irregularity in the area loss values for the pine series. The acceptance limits prescribed by the New York Building Code (unburned area for hardwoods, at least 55 per cent; for softwoods, at least 45 per cent) correspond to loss in area not greater than 45 per cent for hardwoods and 55 per cent for softwoods. The only specimens failing to meet these requirements were the untreated poplar. The untreated pine and oak, and also the treated oak which was known to have nonuniform distribution of treatment, met these requirements. The favorable results in the oak series suggest the effect of density of the material. Additional requirements of

TABLE XIX.—FINAL WEIGHT LOSS IN TIMBER TEST.

NOTE 1.—Values for final weight loss are given as percentages of the weight of the specimen.

NOTE 2.—For the specimens used in the New York crib test, the board numbers, specific gravity values, and absorption of treating salt are the same as for the specimens used in the "special" crib test (see Table IX).

NOTE 3.—The symbols used are as follows: $\bar{X}$ = average, σ = standard deviation, and v = coefficient of variation. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Moisture, per cent	7.3	6.6	7.3	6.1	6.5	6.76	7.3	6.5	6.1	7.6	6.97	6.89
Weight loss, per cent:												
Max.	32.7	29.7	38.9	42.9	38.4	36.52	35.9	27.8	33.7	27.4	24.1	29.78
Min.	27.8	27.8	26.5	27.0	32.3	28.28	28.5	23.9	30.0	14.8	20.8	23.60
$\bar{X}$	30.34	28.50	33.82	35.28	34.86	32.56	31.8	26.1	31.8	20.12	23.26	26.62
σ	1.60	0.65	4.32	5.61	2.17	2.87	3.26	1.44	1.51	4.29	1.38	2.38
v	5.28	2.28	12.78	15.90	6.23	8.82	10.24	5.52	4.75	21.3	5.93	8.94
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Moisture, per cent	10.0	7.0	8.7	7.5	7.6	8.16	8.2	6.8	6.3	10.0	8.45	7.95
Weight loss, per cent:												
Max.	32.8	23.1	28.5	30.4	30.3	29.02	18.7	19.0	24.3	34.4	19.7	23.22
Min.	27.9	20.7	25.6	23.6	28.4	25.24	17.0	17.7	20.4	28.8	16.6	20.10
$\bar{X}$	30.54	21.60	26.80	27.14	29.4	27.09	17.9	18.4	21.48	31.22	17.94	21.39
σ	1.57	0.90	0.95	2.84	0.71	1.39	0.74	0.50	1.44	1.91	1.01	1.12
v	5.14	4.16	3.54	10.46	2.42	5.13	4.13	2.72	6.70	6.11	5.64	5.24
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Moisture, per cent	12.7	6.9	7.9	6.6	6.7	8.16	5.5	6.2	8.1	6.9	7.1	6.76
Weight loss, per cent:												
Max.	29.2	30.6	27.8	23.2	32.7	28.70	61.6	50.5	50.5	67.3	51.3	56.24
Min.	25.2	21.2	22.6	21.5	25.7	23.24	50.8	41.8	41.2	63.3	46.0	48.62
$\bar{X}$	26.34	25.00	24.64	22.56	28.28	25.36	57.06	45.80	46.56	65.12	48.60	52.63
σ	1.48	4.50	1.76	0.59	2.42	2.15	4.08	3.03	3.47	1.59	1.91	2.82
v	5.62	18.0	7.13	2.62	8.55	8.49	7.14	6.61	7.45	2.44	3.94	5.36
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 5.0% CONCENTRATION					
Moisture, per cent	8.4	6.9	6.6	7.3	9.4	7.74	7.2	6.5	7.8	7.4	6.4	7.06
Weight loss, per cent:												
Max.	34.1	18.6	24.1	33.8	20.9	26.30	37.0	30.8	31.2	34.8	29.9	25.80
Min.	28.8	16.4	20.1	28.8	17.2	22.26	29.9	24.0	25.4	22.2	27.5	25.80
$\bar{X}$	31.04	17.60	22.18	31.04	19.28	24.23	33.48	25.80	28.72	26.56	28.27	28.57
σ	2.20	0.72	1.44	1.93	1.40	1 54	2.36	2.54	2.05	4.31	0.87	2.43
v	7.09	4.09	6.50	6.21	7.26	6.35	7.05	9.85	7.15	16.22	3.08	8.52
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Moisture, per cent	11.4	10.3	8.3	9.6	9.7	9.86	8.6	6.5	8.4	7.8	6.9	7.64
Weight loss, per cent:												
Max.	31.9	24.9	26.7	41.5	25.5	30.10	36.8	22.8	27.3	31.2	26.7	28.96
Min.	24.4	20.8	21.3	34.8	21.0	24.46	29.4	21.1	25.1	27.0	20.9	20.70
$\bar{X}$	27.46	23.30	24.00	38.12	23.50	27.28	31.8	21.9	26.06	29.64	23.62	26.60
σ	2.96	1.39	1.79	2.67	1.98	2.16	2.68	0.54	0.71	1.47	1.98	1.48
v	10.76	5.96	7.46	7.01	8.42	7.92	8.42	2.46	2.72	4.95	8.38	5.57

the New York Code limit the duration of flaming to 15 sec. and duration of glowing to 20 sec. The 7.5 and 12.5 per cent concentrations of treatment for the pine and the 12.5 per cent concentration of treatment for the poplar meet these requirements. The limitations of flaming and glowing appear to be more critical than the unburned area requirement, which shows little differentiation between treatments.

TABLE XX.—DURATION OF FLAME IN TIMBER TEST.

NOTE 1.—Values for duration of flame after removal of the burner are given in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table XIX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
Southern Yellow Pine, Untreated							**White Oak, Untreated**					
Max.	5.92	4.25	2.40	4.97	5.95	4.70	4.54	1.92	2.43	3.40	1.35	2.73
Min.	3.71	1.92	1.69	1.04	1.58	1.99	2.25	1.57	1.42	1.12	0.55	1.38
$\bar{X}$	4.24	2.84	2.08	3.43	3.01	3.12	3.24	1.70	1.84	1.90	0.77	1.89
σ	0.82	0.88	0.25	1.30	1.71	0.991	1.01	0.13	0.32	0.85	0.29	0.519
v	19.33	31.00	12.00	37.90	56.80	31.80	31.20	7.66	17.38	44.70	37.65	27.42
Southern Yellow Pine, 2.5% Concentration							**White Oak, 12.5% Concentration**					
Max.	0.37	0.55	1.02	0.38	1.23	0.71	0.47	0.52	0.42	0.63	0.30	0.47
Min.	0.28	0.31	0.31	0.27	0.43	0.32	0.28	0.33	0.20	0.27	0.27	0.27
$\bar{X}$	0.33	0.40	0.57	0.33	0.72	0.47	0.346	0.410	0.314	0.360	0.278	0.340
σ	0.031	0.095	0.300	0.048	0.324	0.160	0.063	0.069	0.080	0.137	0.010	0.072
v	9.40	237.5	52.60	13.57	45.00	34.10	18.20	16.82	25.45	38.05	3.60	21.20
Southern Yellow Pine, 7.5% Concentration							**Yellow Poplar, Untreated**					
Max.	0.16	0.27	0.26	0.27	0.27	0.25	5.09	4.96	4.62	4.87	3.45	4.60
Min.	0.11	0.17	0.13	0.16	0.08	0.13	0.71	2.72	1.87	1.92	1.80	1.80
$\bar{X}$	0.14	0.21	0.19	0.21	0.18	0.19	2.39	3.44	3.44	2.90	2.60	2.95
σ	0.019	0.041	0.040	0.060	0.025	0.037	1.54	0.86	0.93	1.05	0.57	0.990
v	13.58	19.51	21.05	28.60	13.90	19.46	64.40	25.00	27.05	36.20	21.90	33.50
Southern Yellow Pine, 12.5% Concentration							**Yellow Poplar, 5.0% Concentration**					
Max.	0.12	0.09	0.12	0.16	0.08	0.11	0.58	0.34	0.92	0.31	0.32	0.49
Min.	0.07	0	0.10	0.09	0	0.05	0.22	0.27	0.18	0.22	0.20	0.22
$\bar{X}$	0.094	0.030	0.110	0.110	0.054	0.080	0.37	0.31	0.56	0.25	0.24	0.35
σ	0.016	0.034	0.009	0.010	0.033	0.020	0.15	0.03	0.51	0.05	0.05	0.157
v	17.01	113.2	8.18	9.10	61.1	25.0	40.6	9.68	91.1	20.0	20.8	44.85
Southern Yellow Pine, 5.6% Concentration Borax							**Yellow Poplar, 12.5% Concentration**					
Max.	0.22	0.27	0.20	0.19	0.17	0.21	0.11	0.15	0.13	0.12	0.08	0.12
Min.	0.16	0.15	0.15	0.16	0.10	0.14	0.08	0.07	0.10	0.08	0.05	0.08
$\bar{X}$	0.196	0.200	0.176	0.170	0.130	0.170	0.09	0.10	0.12	0.10	0.07	0.10
σ	0.018	0.039	0.014	0.045	0.031	0.029	0.010	0.029	0.010	0.014	0.010	0.015
v	9.18	19.50	7.96	26.45	23.80	17.05	11.1	29.0	8.33	14.0	14.3	15.0

Referring also to the New York Code requirements for the crib test, duration of flaming is limited to 20 sec. and duration of glowing to 30 sec. The 12.5 per cent concentrations of treatment for the pine and poplar meet these requirements. Considering the limitations as a whole, only these two treatments meet all of the New York Code requirements.

COMMENTS ON TEST METHODS AND RESULTS

The reproducibility of test results for this investigation has been discussed with the analysis of the data. The sensitivity of the methods in differentiating degrees of resistance to flammability is indicated by the range of the measured quantities. Except for the timber test, the loss in weight differs over a sufficiently wide range in all tests to distinguish between poorly and well-treated material. The "special" crib test has greater sensitivity than the other tests on the more effective treatments which approximate the practical applications. The maximum temperature and rate of weight loss in the fire tube and "special" crib tests furnish about the same information as the final weight loss. In the timber test, the duration of flaming and glowing provide a more sensitive measure of treatment than the weight loss or area loss. The duration of flaming and glowing is very small for the timber and fire-tube tests on treated materials, with a small range as compared to the two types of crib tests. Good correlation exists between the weight loss after removal of the burner and the total time of continued combustion represented by flaming and glowing in the "special" crib test.

TABLE XXI.—DURATION OF GLOW IN TIMBER TEST.

NOTE 1.—Values for duration of glow after flaming ceased are given in minutes.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table XIX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average
Southern Yellow Pine, Untreated						
Max.	2.13	5.92	2.73	5.25	4.92	4.19
Min.	1.29	0.45	1.57	3.97	0.08	1.47
$\bar{X}$	1.61	2.71	2.13	4.38	2.10	2.59
σ	0.33	1.90	0.42	0.82	1.79	1.052
v	20.5	70.4	19.7	18.72	85.2	40.7
Southern Yellow Pine, 2.5% Concentration						
Max.	0	0	0	0.38	0	0.08
Min.	0	0	0	0.27	0	0.05
$\bar{X}$	0	0	0	0.33	0	0.07
σ	0	0	0	0.036	0	0.007
v	0	0	0	10.90	0	10.0
Southern Yellow Pine, 7.5% Concentration						
Max.	0.02	0	0.04	0.26	0	0.06
Min.	0	0	0	0.16	0	0.03
$\bar{X}$	0.008	0	0.022	0.21	0	0.05
σ	0.007	0	0.010	0.032	0	0.010
v	87.5	0	45.5	152.2	0	20.0
Southern Yellow Pine, 12.5% Concentration						
Max.	0	0.12	0.10	0.19	0.08	0.10
Min.	0	0	0.03	0.11	0	0.03
$\bar{X}$	0	0.05	0.074	0.138	0.016	0.06
σ	0	0.049	0.030	0.028	0.032	0.028
v	0	98.0	40.6	20.25	200.0	46.7
Southern Yellow Pine, 5.6% Concentration Borax						
Max.	2.54	2.85	6.53	3.42	4.33	3.93
Min.	0.67	0.65	1.52	0.83	1.50	1.03
$\bar{X}$	1.856	1.580	3.916	2.260	2.440	2.41
σ	0.695	0.713	1.830	0.842	0.996	1.015
v	37.45	45.1	46.8	37.3	40.8	42.2

Laboratory	C	F	W	B	P	Average
White Oak, Untreated						
Max.	2.50	2.23	2.65	3.65	2.38	2.68
Min.	1.17	0.90	1.42	2.06	0.45	1.20
$\bar{X}$	1.91	1.63	2.08	2.51	1.46	1.92
σ	0.47	0.49	0.42	0.60	0.73	0.542
v	24.6	30.05	20.20	23.9	50.0	28.22
White Oak, 12.5% Concentration						
Max.	0.03	0	0	0.81	0	0.17
Min.	0	0	0	0	0	0
$\bar{X}$	0.006	0	0	0.26	0	0.05
σ	0.012	0	0	0.316	0	0.066
v	200.0	0	0	121.3	0	132.0
Yellow Poplar, Untreated						
Max.	12.5	0.75	1.02	5.17	2.37	4.36
Min.	0.96	0.33	0.02	1.80	0.38	0.70
$\bar{X}$	5.99	0.58	0.67	3.41	1.40	2.41
σ	4.20	0.14	0.19	0.84	0.70	1.215
v	70.2	24.15	28.4	24.6	50.0	50.4
Yellow Poplar, 5.0% Concentration						
Max.	0.03	0	0	0.32	0	0.07
Min.	0	0	0	0.21	0	0.04
$\bar{X}$	0.006	0	0	0.25	0	0.05
σ	0.012	0	0	0.039	0	0.010
v	200.0	0	0	15.6	0	20.0
Yellow Poplar, 12.5% Concentration						
Max.	0	0.10	0	0	0	0.02
Min.	0	0	0	0	0	0
$\bar{X}$	0	0.03	0	0	0	0.01
σ	0	0.041	0	0	0	0.008
v	0	136.5	0	0	0	80.0

Comments on the operation of the tests and difficulties experienced, which were submitted by the cooperating laboratories along with the test data for use in assigning causes of variations, are summarized as follows:

General.—In all of the tests included in this investigation, the adjustment and maintenance of the ignition flames, burners, or furnace, was affected by fluctuations in gas pressure and quality of the gas. Where the gas supply is variable, some form of pressure control seems advisable. The duration of flaming and glowing was difficult to measure with accuracy and subject to varied interpretations as to whether the subsequent blue flame should be included with the yellow flame existing during blazing. The termination of glow on poorly treated material was also difficult to determine.

Fire Tube Test.—The apparatus is rather heavy and bulky and its sensitivity to weight loss may be lowered by friction on the knife-edges. The weight loss may be read accurately to 1 per cent. During the test the charred end of the specimen may drop into the cage, thus changing the relative position of the specimen and the burner flame, and making it difficult for the operator to maintain the burner directly under the specimen and to record flaming or glowing on the piece remaining suspended. Enclosure of the apparatus in a fume hood does not seem necessary because of the protection against side drafts afforded by the tube and the up-draft developed by the burning of the specimen. The length of specimen provides an opportunity for flame spread, but in well-treated material only the lower portion of the specimen is affected.

"Special" Crib Test.—The test provides conditions very easily duplicated. The relative position of the burner flame and specimen is maintained throughout the test without manipulation and, except for untreated material subject to long periods of glow, the charred material remains in place. The entire crib, representing a composite sample, is directly affected by the burner flame with vary-

TABLE XXII.—LOSS OF CROSS-SECTIONAL AREA IN TIMBER TEST.

NOTE 1.—Values for loss of cross-sectional area are expressed as percentages of the original cross-sectional area of the specimen.

NOTE 2.—The data in this table were obtained from the specimens referred to in Table XIX. In each case five specimens were tested.

Laboratory	C	F	W	B	P	Average	C	F	W	B	P	Average
	SOUTHERN YELLOW PINE, UNTREATED						WHITE OAK, UNTREATED					
Max.	51.5	54.7	60.1	64.8	51.6	56.54	48.8	42.2	50.2	53.7	33.3	45.64
Min.	37.0	41.4	39.0	45.3	50.0	42.54	36.4	35.8	43.3	35.5	29.8	36.16
$\bar{X}$	44.78	45.50	49.64	57.08	50.10	49.42	44.86	38.80	47.72	42.97	32.10	41.29
σ	5.49	4.79	7.20	8.12	1.18	5.36	4.30	2.13	2.37	7.30	1.30	3.48
v	12.22	10.51	14.50	14.22	2.36	10.85	9.60	5.50	4.97	17.0	4.05	8.44
	SOUTHERN YELLOW PINE, 2.5% CONCENTRATION						WHITE OAK, 12.5% CONCENTRATION					
Max.	62.3	44.1	45.5	57.6	47.5	51.40	33.3	32.1	42.5	59.3	31.5	39.74
Min.	49.1	41.4	42.3	47.2	43.1	44.62	26.1	29.3	33.8	47.6	22.6	31.88
$\bar{X}$	53.72	42.80	43.97	52.80	45.32	47.72	28.96	30.70	38.83	53.99	28.02	36.10
σ	4.85	1.11	1.12	3.49	1.57	2.43	3.88	0.98	3.27	4.54	4.12	3.36
v	9.03	2.60	2.55	6.60	3.47	5.09	13.4	3.19	8.42	8.40	14.7	9.31
	SOUTHERN YELLOW PINE, 7.5% CONCENTRATION						YELLOW POPLAR, UNTREATED					
Max.	59.7	59.8	48.8	52.7	58.2	55.84	77.8	74.0	74.3	89.0	65.3	76.08
Min.	49.6	49.4	40.0	43.0	50.0	46.40	69.2	66.2	53.0	78.5	56.4	64.66
$\bar{X}$	53.72	54.90	43.54	48.86	50.84	50.37	74.68	69.90	65.58	83.28	61.56	71.00
σ	3.61	4.61	3.52	3.04	3.96	3.75	2.99	2.53	8.38	3.63	3.20	4.15
v	6.71	8.40	8.08	6.22	7.79	7.45	4.00	3.62	12.78	4.36	5.20	5.85
	SOUTHERN YELLOW PINE, 12.5% CONCENTRATION						YELLOW POPLAR, 2.5% CONCENTRATION					
Max.	62.5	47.9	50.0	60.6	44.1	53.02	69.1	53.9	50.9	55.7	46.6	55.24
Min.	54.5	39.4	40.8	48.3	33.3	43.26	53.0	42.1	34.2	42.2	43.1	42.92
$\bar{X}$	58.88	42.80	44.72	54.54	39.98	48.18	57.90	46.70	46.20	48.44	45.50	48.95
σ	3.01	3.23	3.46	5.80	4.20	3.94	5.84	3.94	6.49	5.52	1.84	4.73
v	5.11	7.55	7.73	10.61	10.5	8.17	10.08	8.43	14.01	11.40	4.05	9.66
	SOUTHERN YELLOW PINE, 5.6% CONCENTRATION BORAX						YELLOW POPLAR, 12.5% CONCENTRATION					
Max.	55.5	55.0	51.4	66.4	46.6	54.98	57.8	44.4	49.0	53.2	46.6	50.20
Min.	43.1	48.5	41.6	58.9	36.9	45.80	49.6	39.0	40.7	48.1	35.1	40.50
$\bar{X}$	49.90	51.00	45.83	61.18	41.58	49.90	53.20	40.40	45.42	50.70	39.88	45.92
σ	4.56	2.36	3.86	2.68	3.98	3.49	3.05	2.08	2.85	2.03	4.36	2.87
v	9.13	4.63	8.42	4.38	9.57	7.00	5.73	5.15	6.28	4.00	10.92	6.25

ing intensity from bottom to top. The conditions are more favorable to continued flaming and glowing than in the fire tube test. There is very little friction in the weighing apparatus, and by actual weights of specimens before and after test made by laboratory B, the observations were correct in nearly all cases to within 0.3 per cent. Use of a fume hood does not seem necessary when the shield is employed.

TABLE XXIII.—FLAME SPREAD TESTS.

NOTE 1.—The data in this table are from tests made by laboratory B. Values for specific gravity and moisture content of the specimens for the flame spread tests were the same as for the specimens representing the same treatments in the "special" crib tests performed by laboratory B (see Table IX). In each case five specimens were tested.

NOTE 2.—For information on treatments of test boards, see Table I.

	SOUTHERN YELLOW PINE					WHITE OAK		YELLOW POPLAR		
Treatment concentration, per cent	0	2.5	7.5	12.5	5.6 (borax)	0	12.5	0	5.0	12.5
Board No.	56	53	28	36	47	24	21	25	17	24
TIME OF FLAMING, MIN.										
Max.	4.43	7.75	1.3	0	4.17	5.3	5.75	4.17	4.16	0
Min.	3.87	4.00	0	0	0.58	5.0	0.33	3.0	0.83	0
$\bar{X}$	4.78	5.73	0.41	0	2.54	5.26	3.98	3.7	2.58	0
σ	0.21	1.32	0.56	0	1.48	0.14	1.94	0.42	1.12	0
v	4.40	25.2	136.5	0	58.3	2.66	48.8	11.33	43.4	0
FINAL WEIGHT LOSS, PER CENT										
Max.	82.8	70.0	13.4	9.7	35.9	78.5	72.4	85.0	61.7	13.6
Min.	79.5	47.3	8.1	6.0	12.0	77.1	15.2	82.7	15.2	10.0
$\bar{X}$	80.88	65.32	11.08	7.5	26.5	77.9	60.0	84.36	37.6	11.46
σ	1.1	2.0	1.8	1.26	9.3	0.49	22.4	0.85	17.4	1.2
v	1.36	3.06	16.25	16.80	35.1	0.63	37.3	1.01	46.3	10.47
RATE OF WEIGHT LOSS, PER CENT PER MIN.										
Max.	16.5	11.6	10.0	9.7	7.59	14.95	14.84	21.2	15.43	13.6
Min.	14.92	8.0	5.75	6.0	6.36	12.85	10.38	16.38	6.9	10.0
$\bar{X}$	15.61	9.87	8.09	7.39	7.09	14.24	12.13	18.1	10.23	11.46
σ	0.84	1.36	1.36	1.33	0.53	0.73	1.6	1.8	3.2	1.2
v	5.38	13.78	16.80	18.00	7.48	5.12	13.19	9.95	31.25	10.47

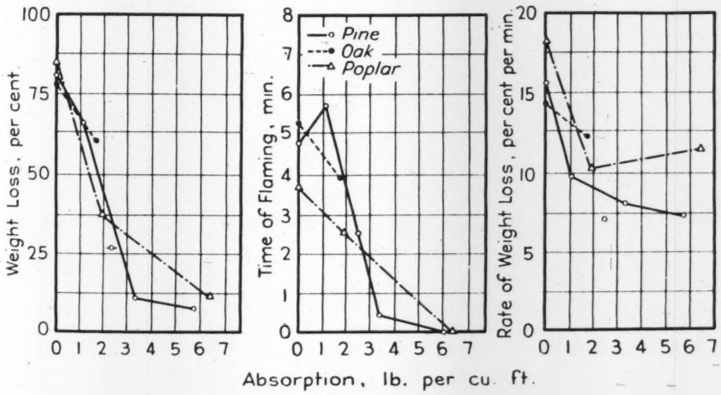

FIG. 20.—Flame Spread Tests.
(Made by laboratory B)

N. Y. Crib Test.—The equipment required is very simple. The test flame is less severe than the above tests, but the type of specimen is favorable to burning, resulting in almost complete consumption for untreated material. While the burner flame is protected by the shield, it is advisable to conduct tests in a fume hood to prevent side drafts which would affect flaming and glowing of the specimen. In well-treated samples only a small area in direct contact with the flame is affected.

Timber Test.—The stabilizing and duplication of furnace conditions gave difficulty to some operators. The removal of the fire brick before placing the specimens also was inconvenient. The transfer of specimens from the furnace to the angle-iron supports required care to avoid affecting the flaming and glowing. Such placement is advisable under a fume hood to avoid side drafts. Difficulty was reported in the determination of the location of the least cross-section before cutting. With an ordinary planimeter, the unburned area selected may be measured to within about 1 or 2 per cent.

Flame Spread Test.—This test conducted by laboratory B has the advantage of simplicity of operation, requiring no special apparatus. No particular difficulties were reported. For untreated and well-treated samples the variations of results were within a small range. For intermediate treatments somewhat greater variation of observations on specimens from the same sample resulted, but the average values of weight loss differentiated between treatments as in most of the other tests (see Fig. 20).

Respectfully submitted on behalf of the subcommittee,

W. J. KREFELD,
Chairman.

REPORT OF COMMITTEE C-7

ON

LIME

Committee C-7 on Lime has held two meetings during the year: on June 26, 1940, in Atlantic City, N. J., and on March 3, 1941, in Washington, D. C. A special meeting of the Advisory Committee was held on October 9, 1940, in Washington, D. C.

Several changes in committee personnel have occurred since the latest report to the Society. At present the total membership is 49, classified as 24 producers, 7 consumers, and 18 general interest members.

During the year a complete reorganization of all subcommittees was effected. The purpose of the reorganization was to reduce the size of the subcommittees and the overlapping of membership thereon to as great an extent as possible, with the thought that more effective work could be accomplished. Progress in this direction has been made.

Amendment to By-laws.—The following amendment to Article V, Section 3, of the committee's by-laws was adopted to make this section consistent with the Society's Regulations Governing Standing Committees, with respect to voting on standards and tentative standards:

> "Recommendations affecting standards or tentative standards shall be agreed upon only at meetings called for that purpose. An affirmative vote amounting to two-thirds of those voting shall be required on all recommendations affecting standards and tentative standards, subject to confirmation by letter ballot of the entire committee on which an affirmative vote amounting to two-thirds of those voting shall be required. 'Those voting' consists of those casting either an affirmative or a negative vote. 'Not voting' ballots are those returned so marked."

This amendment to the by-laws of Committee C-7 has been submitted to letter ballot of the committee which consists of 49 members; 39 members returned their ballots, of whom 36 have voted affirmatively, 2 negatively, and 1 member returned his ballot marked "not voting."

Evaluating Cubic Foot Quantities of Mortar Materials.—For the purpose of standardizing test conditions in connection with investigations on mortars so that the results from different sources might be correlated more closely, the committee adopted the following values for cubic foot quantities of the usual ingredients in mortars:

Lime hydrate solids[a]	50 lb. per cu. ft.
Portland cement	94 lb. per cu. ft.
Sand	80 lb. per cu. ft.

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[a] A 50 lb. bag of hydrated lime shall be considered as 1 cu. ft. of material. When quick lime putty is used 50 lb. of solids in sufficient putty shall be considered 1 cu. ft. of lime hydrate solids.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Structural Lime (J. A. Murray, chairman):

Study of Hydrated Limes.—In view of recent changes made by the lime industry in the processing of hydrated limes, an attempt was made by Subcommittee II to obtain information both on the service performance of these specially processed hydrates and on laboratory tests which might be used as a basis for specifications. An insufficient amount of data for this purpose has been made available up to the present time. Therefore, the subcommittee is continuing its investigation looking toward a future revision of the Standard Specifications for Hydrated Lime for Structural Purposes (C 6 – 31).

Special Subcommittee on Lime in Mortar.—With the objective of assembling, correlating, and studying the existing data on lime in mortar, and developing as much new information as possible, a Special Subcommittee on Lime in Mortar was appointed to function under the general direction of Subcommittee II. A preliminary report was prepared by this special subcommittee which included a discussion of the contribution of lime to the properties desired in mortars, and a brief résumé of the present knowledge of the subject gained from studies made by investigators in the United States and foreign countries. A rather extensive bibliography on mortars, masonry, and related subjects also was compiled and presented as a part of the report of the special subcommittee.

Mortar Strength Tests.—Subcommittees II and V are collaborating in a study of methods for determining the strength of mortars and its relation to the strength developed in masonry. New methods for testing the mortar strength and the effects of different curing conditions on the results obtained by the cube test are to be investigated.

Subcommittee V on Methods of Test (J. F. Carle, chairman):

Methods for Determining Available Lime.—A series of tests on the various sugar and other methods for determining "available lime" is being conducted for the purpose of standardizing on a simple and fairly accurate procedure which can be used by the producer and consumer in evaluating lime products for various uses.

Methods of Analysis for Arsenic, Fluorine, and Lead.—Subcommittee V has under way the development of analytical methods for determining the amount of arsenic, fluorine, and lead possibly present in lime used in the manufacture of food products, such as baking powder.

Methods for Slaking Quicklimes.—Data are being collected by Subcommittee V on the various methods used in slaking quicklime on the job for the purpose of duplicating these conditions in the laboratory and preparing a laboratory procedure on the slaking process. Information regarding the size and shape of the putty box, depth of putty, atmospheric temperature and other weather conditions, amounts of lime and water used, type of lime, initial temperature of the water, temperatures at different stages in the slaking process, time required, and the nature of the resulting putty, is being furnished by members of Committee C-7.

Settling Rates of Lime Products.—Since the respective settling rates of different types of quicklime and hydrated lime are of major importance in a number of chemical and industrial processes using lime, Subcommittee V is investigating the existing methods prior to the development of a procedure which can be presented to Committee C-7 for consideration.

Subcommittee VIII on Nomenclature and Definitions (W. V. Brumbaugh, chairman):

A series of 23 proposed definitions covering lime and lime products, and terms relating to lime, has been prepared by Subcommittee VIII and transmitted to all members of Committee C-7 for comment. It is expected that agreement can be reached on some of these definitions during the next year.

RECOMMENDATIONS AFFECTING STANDARDS

A number of proposed revisions and additions to the committee's Tentative Methods of Physical Tests of Quicklime and Hydrated Lime (C 110 – 38 T) are under consideration, but in view of the fact that the work necessary in connection with these revisions will not be completed in the near future, the committee recommends that the methods be continued as tentative for another year.

In the absence of specific action during the year, the committee recommends continuing without revision the Tentative Specifications for Hydraulic Hydrated Lime for Structural Purposes (C 141 – 38 T).

Although no comments have been received during the year relative to the tentative revision of the Standard Specifications for Quicklime and Hydrated Lime for Silica Brick Manufacture (C 49 – 24) which was presented in the 1940 report, the committee recommends that this revision be continued as tentative for another year.

During the year the committee conducted a canvass of leading varnish producers to determine whether the Standard Specifications for Hydrated Lime for Varnish Manufacture (C 47 – 27) needed revision. The results of the survey showed that practically all of the manufacturers who replied are following the original specifications in the purchase of hydrated lime for this purpose. The committee therefore recommends that these specifications be continued as standard without revision.

This report has been submitted to letter ballot of the committee, which consists of 49 members; 36 members returned their ballots, of whom 32 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

NATHAN C. ROCKWOOD,
Chairman.

W. VERNON BRUMBAUGH,
Secretary

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-7 presented to the Society through Committee E-10 on Standards a tentative revision of the Standard Definitions of Terms Relating to Lime (C 51 – 39). This tentative revision, in the form of new Tentative Definitions of Terms Relating to Lime (C 51 – 41 T), was accepted by Committee E-10 on October 23, 1941, and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 265.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES

Committee C-8 on Refractories held two meetings during the year: one on September 5, 1940, at the Summit Hotel, near Uniontown, Pa., in conjunction with the Refractories Division of the American Ceramic Society, and the other on March 6, 1941, at Washington, D. C., with the spring group meetings of the A.S.T.M.

The committee was active during the year. The Editorial Subcommittee prepared a new edition of the Manual of A.S.T.M. Standards on Refractory Materials which was published in January, 1941. In order to include as many standards as possible in the new manual, the following recommendations, resulting from actions of the committee at its meeting on September 5, 1940, were submitted to the Society through Committee E-10 on Standards:

Tentative Revisions of Standard:

Method of Test for Pyrometric Cone Equivalent of Refractory Materials (C 24 – 35),

Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 36),

Method of Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 40), and

Method of Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 40).

Withdrawal of Standard:

Definitions of Fireclay and Alumina-Diaspore Refractories (C 27 – 39), and publication as tentative of the revised Classification of Fireclay Refractories (C 27 – 40 T).

These recommendations were accepted[1] by Committee E-10 on November 5, 1940, and the tentative revisions and revised tentative definitions appear in the 1941 edition of the Manual of A.S.T.M. Standards on Refractory Materials and in the 1940 Supplement to Book of A.S.T.M. Standards, Part II.

In 1939 the committee first presented to the Society a classification of certain refractory products in lieu of definitions. Classifications have been handled to date by the Subcommittee on Nomenclature, but as they are not definitions it seemed desirable to establish a new subcommittee to handle them. A new Subcommittee IX on Classifications (E. H. Van Schoick, chairman) was accordingly formed.

A new Section H on Consistency and Plasticity (P. G. Herold, chairman), under Subcommittee III on Tests was established to work on test methods useful for evaluating fireclay plastic refractories, cold-set mortars, and similar refractory materials.

I. REVISION OF TENTATIVE STANDARD

Tentative Classification of Insulating Block and Insulating Fire Brick (*C 155 – 40 T*).[2]—The committee recommends that this Tentative Classification C 155 be revised to indicate that it covers insulating back-up block in order to differentiate the material covered from pipe insulation under the jurisdiction of

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee C-8 reported results of the letter ballot vote as follows: Of a total voting membership of 36, 32 members returned their ballots, of whom 30 voted affirmatively, 0 negatively, and 2 marked their ballot "not voting."

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 221.

Committee C-16 on Thermal Insulating Materials. The committee also recommends, after careful consideration and after receiving comments from industry, that the maximum bulk densities be revised. The committee believes that the proposed revised values are in line with present manufacturing practice, and that further revisions of the classification along these lines are not likely to occur within the next three years. It is accordingly recommended that this classification be revised as follows and continued as tentative:

Title.—Change the title to read as follows by the addition of the italicized words:

Tentative Classification of Insulating *Back-Up* Block and Insulating Fire Brick.

Section 2.—Insert the word "back-up" between "insulating" and "block" in the title of this section and in the italicized heading of Paragraph 2; change the value of the bulk density from "35" to "30" lb. per cu. ft.

Table I.—Change the values for the maximum bulk densities to read as follows by the addition of the italicized figures and the omission of those in brackets:

Group 16	[38] *34* lb. per cu. ft.
Group 20	[46] *40* lb. per cu. ft.
Group 23	[53] *48* lb. per cu. ft.
Group 26	[58] *52* lb. per cu. ft.
Group 28	[62] *60* lb. per cu. ft.

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Committee C-8 recommends that the twelve tentative standards referred to below, consisting of five methods of test, four specifications, the methods of chemical analysis, definitions, and classification, be approved for reference to letter ballot of the Society for adoption as standard for the reasons given:

Specifications for Refractories.—Previous to 1940 there were three standard specifications for refractories: namely, Standard C 63 relating to fireclay refractories for malleable furnaces, Standard C 64 for stationary boiler service, and Standard C 106 for incinerators. As these standards had not been changed appreciably for a number of years, and because a new classification of fireclay refractories was being presented, the committee decided to rewrite the specifications, incorporating the new classification in them, and also improving the presentation of the subject matter. New separate specifications were written for refractories for heavy duty service and moderate duty service boilers. These four new specifications were published as tentative in 1940. Since no criticism has been received, the committee believes they should be adopted as standard:

Tentative Specifications for:[3]

Refractories for Heavy Duty Stationary Boiler Service (C 64 - 40 T),

Refractories for Moderate Duty Stationary Boiler Service (C 153 - 40 T),

Refractories for Incinerators (C 106 - 40 T), and

Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (C 63 - 40 T).

Methods of Test.—Since no criticisms of the following tentative methods have been received, it is recommended that they be adopted as standard without revision:

Tentative Methods of:

Testing Fireclay Refractories Under Load at High Temperatures (C 16 - 39 T),[4]

Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (C 20 - 39 T),[4]

Testing Insulating Fire Brick, (Compressive Strength, Flexural Strength, and Permanent Linear Change after Heating) (C 93 - 39 T),[4]

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 202, 206, 208, 199.

[4] 1939 Book of A.S.T.M. Standards, Part II, pp. 958, 963, 954, 966.

Test for Size and Bulk Density of Refractory Brick (C 134 - 39 T)[4] and

Test for Warpage of Refractory Brick and Tile (C 154 - 40 T).[5]

Method C 154 had previously been published as part of Method C 134 - 38 T, which method was revised a year later. In the revision, the description of the warpage test was inadvertently omitted. Although not in the form in which it is now published, Method C 154 was originally issued in 1938, and, as it has been in constant use by the industry since that time, it now merits adoption as standard.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE STANDARD			
Classification of Insulating Block and Insulating Fire Brick (C 155 - 40 T)	31	0	2
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARDS			
Testing Fireclay Refractories Under Load Test at High Temperatures (C 16 - 39 T)	30	0	3
Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (C 20 - 39 T)	31	0	2
Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change) (C 93 - 39 T)	31	0	2
Test for Size and Bulk Density of Refractory Brick (C 134 - 39 T)	32	0	1
Chemical Analysis of Chrome Ore and Chrome Refractories (C 18 - 40 T)	28	0	5
Classification of Fireclay Refractories (C 27 - 40 T)	30	1	2
Definitions of Terms Relating to Refractories (C 71 - 40 T)[7]	32	0	1
Test for Warpage of Refractory Brick and Tile (C 154 - 40 T)	33	0	0
Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 40 T)	29	1	3
Spec. for Refractories for Moderate Duty Stationary Boiler Service (C 153 - 40 T)	29	1	3
Spec. for Refractories for Incinerators (C 106 - 40 T)	28	1	4
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 40 T)	28	1	4
III. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARDS			
Spec. for Ground Fire Clay (C 105 - 36)	29	0	4
Symbols for Heat Transmission (C 108 - 39)	33	0	0

Methods of Chemical Analysis.—The Tentative Methods of Chemical Analysis of Chrome Ore and Chrome Refractories (C 18 - 40 T)[6] are now in general use in the refractories industry. No criticism has been received from either producers or consumers of chrome refractories since their publication as tentative in 1940. The committee believes they should be adopted as standard without revision and incorporated in the Standard Methods of Chemical Analysis of Refractory Materials (C 18 - 39).

Definitions.—No criticism has been received on the definitions of the terms abrasion, corrosion, erosion, and slagging which were published as tentative in 1940. The committee, therefore, is proposing that these terms as they appear in the Tentative Definitions of Terms Relating to Refractories (C 71 - 40 T)[6] be adopted as standard,[7] for incorporation in the Standard Definitions of Terms Relating to Refractories (C 71 - 36).

Classification.—Prior to 1939, Committee C-8 had prepared the Standard Definitions of Fireclay Refractories (C 27 - 37). In 1940, the committee presented a Tentative Classification of Fireclay Refractories (C 27 - 40 T) rather than definitions. In this new classification, high and intermediate heat duty fireclay brick were classified according to refractoriness as measured by P.C.E., *or* by means of their behavior in the load test, and the classification of moderate heat duty fireclay brick was removed. When this tentative classification was accepted by the Society in June, 1940, the committee, through a misunderstanding, failed to request that the standard definitions of super duty fireclay brick and of alumina-diaspore refractories of C 27 – 39 be transferred to the new classification so that these products and high heat duty fireclay brick, intermediate heat duty fireclay brick, and low heat duty fireclay brick would be in the same standard. This request was transmitted to Committee E-10 on Standards, which accepted the recommendation on November 5, 1940. The committee now desires to have the Tentative Classification of Fireclay Refractories (C 27 – 40 T)[8] adopted as standard.

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[5] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 218.

[6] 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 211, 222.

[7] The recommendation for adoption as standard of these tentative definitions failed of approval at the annual meeting of the Society, see Summary of Proceedings, p. 34.

III. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Ground Fire Clay (C 105 – 36).—The committee recommends that the revision[9] of these specifications published as tentative in 1940 be approved for reference to letter ballot of the Society for adoption as standard.

Standard Symbols for Heat Transmission (C 108 – 39).—The committee also recommends that the revision[10] of the symbols for heat transmission, published as tentative since 1939, be approved for reference to letter ballot of the Society for adoption as standard.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 37 members; 33 members returned their ballots with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (L. C. Hewitt, chairman), completed the following two new industrial surveys during the year.

Industrial Survey of Refractory Service Conditions in the Portland Cement Industry, and

Industrial Survey of Refractory Service Conditions in Stationary Steam Boilers.

Both of these have been accepted and appear in the 1941 refractories manual.[11]

The subcommittee is working on surveys of refractory service conditions in zinc smelting and in iron cupolas.

Subcommittee II on Research (R. S. Bradley, chairman) has studied the subject of the rate of application of load to be employed when hydraulic testing machines are used to determine cold crushing strength and modulus of rupture of refractories. The findings have been incorporated as editorial changes in Paragraphs 5 (*d*) and 7 (*c*) of the Standard Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (C 133 – 39).

Subcommittee III on Tests (F. A. Harvey, chairman) is composed of the chairmen of the several sections described below. The activity of the subcommittee has already been brought out in the foregoing recommendations on standards and is further amplified in the following discussion of the work of the individual sections:

Section A on Load was temporarily discontinued.

Section B on Spalling (R. E. Birch, chairman) studied a new style of backup insulation for panels during the reheat portion of the spalling test. The object of this work was to devise a means of insulating the panels to effect a uniform heat distribution at the inner face of the panel and the insulation. As a result, an even spalling takes place over the entire surface of the panel rather than a more or less concentrated action along the center vertical section. As a result of this work, the section recommended last year tentative revisions in the three Standard Methods C 38 – 36, C 107 – 36, and C 122 – 37 as mentioned earlier in this report. These revisions are now being recommended for adoption as standard.

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[8] 1940 Supplement to Book of A.S.T.M. Standards Part II, p. 219.

[9] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 340.

[10] 1939 Book of A.S.T.M. Standards, Part II, p. 1181, or 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 343.

[11] "Manual of A.S.T.M. Standards on Refractory Materials," pp. 150, 156 (1941), issued as a separate publication.

Section C on Temperature (R. B. Sosman, chairman) recommended slight modifications in the size and shape of the pyrometric test cones. This recommendation, as mentioned earlier in this report, was accepted by Committee E-10 on Standards as a tentative revision of Method C 24 – 35.

The section now has under way a comparative study of gas-fired and electric furnaces for determining the P.C.E. of refractory materials. It is also studying the testing of plastic fire clays, particularly those that bloat in the standard P.C.E. test. The study is concerned with calcining the clays prior to making the test cones and in calcining the test cones themselves before making the P.C.E. test.

Section D on Slagging (C. E. Bales, chairman) has devoted its attention to studying the literature and preparing a bibliography for the period 1932 to 1940. The last previous comprehensive bibliography was prepared in 1932.

Section E on Analysis (L. J. Trostel, chairman) has recommended the adoption as standard of the Tentative Methods of Chemical Analysis of Chrome Ore and Chrome Refractories (C 18 – 40 T).

Section F on Tests of Refractory Insulation (W. R. Kerr, chairman) has continued to collect data on the classification of insulating block and insulating fire brick. As a result, a recommendation was made in March, 1941, to revise the Tentative Classification of Insulating Block and Insulating Fire Brick (C 155 – 40 T). The revision, given earlier in this report, involves an editorial change and a lowering of the permissible maximum bulk density for the various group identifications of insulating fire brick. These changes are in line with the products now marketed by manufacturers of these materials.

Section G on Porosity and Permanent Volume Change (C. E. Fulton, chairman) has recommended the adoption as standard of the Tentative Methods of Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (C 20 – 39 T) and Test for Size and Bulk Density of Refractory Brick (C 134 – 39 T).

Subcommittee IV on Heat Transfer (R. H. Heilman, chairman) is continuing to cooperate with the National Bureau of Standards on the development of a standard apparatus to determine thermal conductivity of refractory materials at high temperatures. Mr. M. S. Van Dusen of the Bureau reported to Committee C-8 at the March 6, 1941, meeting in Washington, D. C., that progress is being made, but no definite recommendations are possible at this time.

Two laboratories are making a comparative study of the thermal conductivity of fireclay refractories. One laboratory is using the hot-plate and the other the water-flow calorimeter method, but both make use of Globar elements for obtaining the necessary high temperatures. The study has not progressed far enough to make a formal report to the committee.

Subcommittee V on Precision and Tolerances (R. A. Heindl, chairman) has been attempting to prepare a procedure for statistical methods pertaining to the analysis of refractory test data. The recommended method published in the 1937 Refractories Manual had many shortcomings and, so far as could be learned, very few people made use of the procedure. One reason for this was that the method required data obtained from an appreciable number of specimens; so its use was quite limited unless a series of tests was conducted—a costly procedure not applicable to routine work. The recommended method was omitted from the 1941 manual, and reference is now made to the methods and practices outlined in the A.S.T.M. Manual on Presentation of Data, Third Printing, August, 1940.

Subcommittee VI on Nomenclature (M. E. Holmes, chairman) recommends the adoption as standard of the Tentative Classification of Fireclay Refractories (C 27 - 40 T).

Subcommittee VII on Specifications (G. A. Bole, chairman) has recommended the adoption as standard of the tentative revision of the Standard Specifications for Ground Fire Clay (C 105 - 36) and also the adoption as standard of the four tentative specifications for refractories as mentioned earlier in this report.

The subcommittee is working on specifications for fireclay plastic refractories for boiler furnaces and incinerator service. It is also considering specifications for ground fire clays for patching and daubing mixes, and specifications for air-setting mortars.

Subcommittee VIII, Editorial (L. J. Trostel, chairman) compiled a new edition of the Manual of A.S.T.M. Standards on Refractory Materials during the year. This includes specifications; classifications; methods of testing; definitions; recommended procedure for calculating heat losses through furnace walls; a list of standard samples of refractory materials for chemical analysis; and industrial surveys of refractory service conditions in open-hearth practice, malleable iron industry, copper industry, lead industry, by-product coke ovens, lime burning industry, glass industry, portland cement industry, and stationary steam boilers.

This report has been submitted to letter ballot of the committee which consists of 37 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. SULLIVAN,
Chairman.

L. J. TROSTEL,
Vice-Chairman.

S. M. PHELPS,
Secretary.

REPORT OF COMMITTEE C-9

ON

CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates held three meetings during the year: two in Washington, D. C., on December 7, 1940, and on March 7, 1941, respectively, and one during the annual meeting of the Society in Chicago, Ill.

The Sanford E. Thompson Award this year was won by W. T. Thomson for his paper on "A Method of Measuring Thermal Diffusivity and Conductivity of Stone and Concrete,"[1] which was presented at the 1940 annual meeting of the Society. This is the second Sanford E. Thompson Award.

I. TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Ready Mixed Concrete (C 94 – 38).[2]—The committee submits for publication as tentative a revision of these standard specifications in the form of new proposed Tentative Specifications for Ready Mixed Concrete as appended hereto[3] which were prepared by Subcommittee XVIII on Ready Mixed Concrete (R. B. Young, chairman). The proposed revised specifications recognize developments which have taken place since the adoption of the Standard Specifications C 94 – 38, which are to be continued as standard.

Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39).[4]—The committee recommends, in cooperation with Committee D-4 on Road and Paving Materials, a tentative revision providing for the testing of a wider range in gradings of aggregates in this standard method, which is under the joint jurisdiction of Committees C-9 and D-4. The details of the proposed tentative revision, as indicated below, have been prepared by Subcommittee IX on Specifications and Methods of Tests of Aggregates (H. F. Clemmer, chairman) in the light of experience with this method of test:

Section 3 (b).—Change from its present form: namely,

> (*b*) An abrasive charge of 12 spheres weighing 5000 ± 25 g. shall be used with the grading A described in Section 4, and a charge of 11 spheres weighing 4583 ± 25 g. shall be used with the grading B described in Section 4.

to read as follows:

> (*b*) The abrasive charge, depending upon the grading of the test sample as described in Section 4, shall be as follows:

Grading	Number of Spheres	Weight of Charge, g.
A	12	5000 ± 25
B	11	4584 ± 25
C	8	3330 ± 20
D	6	2500 ± 15

Section 4.—Change from its present form: namely,

> 4. The test sample shall consist of 5000 g. of clean, dry aggregate and shall conform to either of the following gradings. The grading used shall be that most nearly representing the aggregate furnished for the work.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1073 (1940).

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 314.

[3] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 321.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 288.

Sieve Size (Square Openings)		Grading *A*	Grading *B*
Passing	Retained on	Weight, g.	Weight, g.
1½ in.	1 in.	1250	0
1 in.	¾ in.	1250	0
¾ in.	½ in.	1250	2500
½ in.	⅜ in.	1250	2500

to read as follows:

4. The test sample shall consist of 5000 g. of clean, dry aggregate and shall conform to one of the following gradings. The grading used shall be that most nearly representing the aggregate furnished for the work.

Sieve Size (Square Openings)		Weight, g.			
Passing	Retained on	Grading A	Grading B	Grading C	Grading D
1½ in.	1 in.	1250	. . . .	. . . .	. . . .
1 in.	¾ in.	1250	. . . .	. . . .	. . . .
¾ in.	½ in.	1250	2500	. . . .	. . . .
½ in.	⅜ in.	1250	2500	. . . .	. . . .
⅜ in.	No. 3	. . . .	. . . .	2500	. . . .
No. 3	No. 4	. . . .	. . . .	2500	. . . .
No. 4	No. 8	. . . .	. . . .	. . . .	5000

Explanatory Note.—The need for a wider range of gradings for use in the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39) has been met in part by the revisions presented this year by Committees C-9 and D-4. The new gradings C and D provide, in particular, for tests of the finer sizes used in concrete and in bituminous surface treatments. However, a further expansion in the range of gradings is needed to permit of tests of larger sizes of concrete aggregates and of aggregates such as are required for penetration macadam, water-bound macadam and other types of construction utilizing the coarser materials.

Gradings A and B, specified in the method as first adopted by the Society in 1937, require abrasive charges of 12 and 11 steel or cast iron spheres, approximately 1⅞ in. in diameter, respectively. Research leading to the development of the test method showed these charges and gradings to give substantially the same results for the same materials. Similarly, the new gradings C and D requiring 8 and 6 of the same spheres have been demonstrated to give approximately the same results. The data for these four gradings indicate the abrasive charges required for similar losses in tests of material from the same source to be a logarithmic function of the surface area of the test sample.

Based on the relationship referred to, the committees proposed at their spring meetings to include requirements for a coarser grading consisting of 2500 g. of 2½- to 2-in. material and 2500 g. of 2- to 1½-in. material tested with an abrasive charge of 15 of the steel or cast iron spheres. Subsequent tests showed the losses thus obtained to be, with minor exceptions, considerably lower than those found for the four gradings and charges specified in the proposed revised test method. Also tests made with more than 15 spheres as the abrasive charge failed to show proportionate increases in loss. Accordingly, the recommendation of the coarser grading was withdrawn at the June meeting.

In withdrawing that recommendation the committees instructed the subcommittees having the test method in charge to prepare the preceding statement in explanation of the basis for the revisions and to point to the need for further investigation.

Standard Specifications for Concrete Aggregates (*C 33 – 40*).[5]—The committee recommends for publication as tentative the following revision of this standard which has also been prepared by Subcommittee IX in the interest of clarification:

Section 4 (*a*).—Omit the parentheses around the figures under the column in the table headed "Percentage Passing," and change the Note following this section from its present form: namely,

Note.—Figures in parentheses are suggested as limiting percentages but they may be altered within these limits to suit local conditions.

to read as follows:

Note.—Attention is called to the fact that the relatively wide range in grading which is shown in the above table should be permitted only when it is economically impracticable to obtain materials meeting more restrictive requirements. The most desirable grading will depend upon the type of work and the class of concrete. For the leaner mixes, or when a small size coarse aggregate is used, in cases where the degree of workability is important, it is desirable to further restrict the allowable ranges in sizes shown so as to insure a grading approaching the maximum percentage passing each sieve. On the other hand, for the richer mixes, in the interests of maximum strength and economy, a grading as coarse as is consistent with the requirements for workability should be specified. However, in no case should a range in grading be required more restrictive on any one sieve than indicated below:

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[5] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 84.

No. 16 (1190-micron) sieve	20 per cent
No. 50 (297-micron) sieve.........	15 per cent
No. 100 (149-micron) sieve.........	5 per cent

Section 4 (b).—Change the italicized heading of this paragraph from "Fineness Modulus" to read "Uniformity of Grading."

Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (C 87 – 39).[6] The committee recommends for publication as tentative the following revision of this standard method which has also been prepared by Subcommittee IX as the result of experience with this method of test:

Section 3 (b).—Add the following as a new fifth sentence in this paragraph:

> If more than two trials must be made to obtain a flow of 100 ± 5, the mortar should be considered as a trial mortar and the test specimens prepared from a new batch.

II. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The committee recommends that the following three tentative standards, which have stood for two years or more without revision, be continued as tentative without revision for the reasons given:

Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 39 T).[7]—While this tentative method of test is extensively used, it has not yet been established as a generally accepted method of test. It is the committee's judgment that more time must be allowed for trial.[8]

Tentative Method of Test for Soundness of Aggregates by Freezing and Thawing (C 137 – 38 T).—While this tentative method of test has done much to standardize freezing-and-thawing tests, there has not yet been sufficient use of it to warrant its adoption as standard.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
TENTATIVE REVISIONS OF STANDARDS			
Spec. for Ready Mixed Concrete (C 94 – 39)..........	59	0	2
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39)..............	56	0	5
Spec. for Concrete Aggregates (C 33 – 40):			
Note following the grading table in Paragraph 4 (*a*) ..	61	0	6
Paragraph 4 (*b*) change title from "Fineness Modulus" to "Uniformity of Grading"......................	61	3	2
Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (C 87 – 39).....	60	0	7

Tentative Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125 – 36 T).—Conflicting points of view have not yet been sufficiently reconciled with respect to definitions for crushed gravel and blast furnace slag to warrant the adoption of these two definitions as standard.

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[6] 1939 Book of A.S.T.M. Standards, Part II, p. 304.

[7] A revision of this method was accepted by action of the Society at the annual meeting as recorded in the Summary of Proceedings, see p. 35. This action was approved by subsequent letter ballot of the committee which consists of 74 members; 61 members returned their ballots, of whom 52 voted affirmatively, 0 negatively, and 9 members marked their ballots "not voting."

[8] See Editorial Note, p. 289.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 74 members, with the results shown in Table I.

This report has been submitted to letter ballot of the committee which consists of 74 members; 59 members have returned their ballots, of whom 58 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

R. R. LITEHISER,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-9 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative Method of Test for:
- Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 41 T),

Tentative Revisions of Standard:
- Method of Making and Storing Compression Test Specimens of Concrete in the Field (C 31 – 39),
- Method of Test for Compressive Strength of Concrete (C 39 – 39),
- Method of Securing Specimens of Hardened Concrete from the Structure (C 42 – 39),
- Method of Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (C 116 – 39),
- Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 39),
- Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 39), and
- Specifications for Lightweight Aggregate for Concrete (C 130 – 39).

These recommendations were accepted by Committee E-10 on August 25, 1941, and the revised tentative method and tentative revisions appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, pp. 327, and 421 to 424, inclusive.

REPORT OF COMMITTEE C-11

ON

GYPSUM

Committee C-11 on Gypsum held one meeting during the past year at the Mayflower Hotel, Washington, D. C., on March 6, 1941, in conjunction with the spring group meetings of the Society.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Structural Gypsum Products (L. S. Wells, chairman).—This subcommittee reviewed the Federal Specification for Gypsum Partition Tile (SS-T-316) and has revised accordingly the A.S.T.M. Specifications for Gypsum Partition Tile or Block (C 52 - 33).[1] The revised specifications, as appended hereto,[2] are being recommended for adoption as standard.

Subcommittee IV on Testing Methods (C. K. Roos, chairman).—This subcommittee is continuing its study of the ammonium acetate method for determining the purity of gypsum and calcined gypsum. A method suggested by B. E. Kester, who has been working with L. S. Wells and W. F. Clarke of the National Bureau of Standards on this method, is included in an appendix to this report.

I. REVISION OF TENTATIVE STANDARD

Tentative Definitions of Terms Relating to Gypsum (C 11 - 40 T).[3]—For the sake of simplicity, the committee recommends that the present definition of perforated gypsum lath be revised to read as follows and continued as tentative:

> *Perforated Gypsum Lath.*—A gypsum lath with perforations not less than $\frac{3}{4}$ in. in diameter, with one perforation for not more than each 16 sq. in. of lath.

In accordance with the recommendations of Committee E-8 on Nomenclature and Definitions, Committee C-11 recommends the deletion of the definition of the term "gypsum plaster board" as this definition is now unnecessary in view of the changes being recommended later in this report in the definitions of gypsum lath, gypsum sheathing board, and gypsum wall board.

II. REVISIONS OF STANDARDS

Standard Specifications for Gypsum (C 22 - 25).[4]—In these specifications Section 2 on Forms and Sizes, describes four forms of gypsum and four sizes. The secretary, at the request of the committee, submitted this section to the industry for comment and, in accordance with the comments received, the committee recommends for immediate adoption the following revision in Standard Specifications C 22 and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this change may be referred to letter ballot of the Society:

Section 2.—Change the title from "Forms and Sizes" to read "Size" and change this section from its present form: namely,

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 79.

[2] These specifications were adopted as standard by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 7.

[3] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 179.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 63.

2. Gypsum may be in any of the following forms and sizes:

(*a*) *Run-of-Mine Gypsum.*—The form in which it comes from the mine or quarry.

(*b*) *Crushed Gypsum.*—Run-of-mine gypsum further reduced so that all of it passes a 3-in. ring, and not more than 25 per cent passes a No. 100 (149-micron) sieve.

(*c*) *Sized Gypsum.*—Gypsum, all of which shall pass a 1½-in. ring, and not more than 10 per cent of which shall pass a ¼-in. sieve.

(*d*) *Ground Gypsum.*—Gypsum further reduced for specific uses. Ground gypsum may be in any of the following standard forms:

No. 1.—All passing a No. 14 (1410-micron) sieve, and not less than 85 per cent passing a No. 100 (149-micron) sieve.

No. 2.—All passing a No. 14 (1410-micron) sieve, and not less than 60 nor more than 85 per cent passing a No. 100 (149-micron) sieve.

No. 3.—All passing a No. 8 (2380-micron) sieve, and not less than 40 nor more than 60 per cent passing a No. 100 (149-micron) sieve.

No. 4.—All passing a No. 100 (149-micron) sieve.

to read as follows:

2. Gypsum may be crushed and ground to the size specified by the purchaser.

Standard Specifications for Gypsum Partition Tile or Block (*C 52 – 33*).[1]—The committee has been considering the revision of these specifications for several years because of several obsolete sections in the specification, but since the Federal Specifications Board was engaged in the preparation of a federal specification for gypsum partition tile, action was withheld until the federal specification was approved. This specification has now been promulgated, and Committee C-11 has revised the Standard Specifications for Gypsum Partition Tile or Block (C 52 – 33) so that they are in agreement with the Federal Specification for Gypsum Partition Tile (SS-T-316). The revised specifications appended hereto[2] are accordingly recommended for immediate adoption to replace the present standard. The committee accordingly asks for a favorable nine-tenths vote at the annual meeting in order that the revised specifications may be referred to letter ballot of the Society.

Standard Methods of Testing Gypsum and Gypsum Products (*C 26 – 40*).[5]—In Sections 37 and 38 of Standard Methods C 26, a procedure is outlined for the determination of the percentage of absorption and rate of absorption of gypsum partition tile. The revised Specifications for Gypsum Partition Tile or Block,[2] which the committee is recommending for adoption as standard at this meeting, do not contain any reference to percentage of absorption or rate of absorption. It is not felt necessary to include requirements for absorption because the specifications prescribe a minimum compressive strength when saturated and also specify that the surfaces of the tile shall be such that they afford a suitable bond with plaster. The committee recommends the immediate deletion of Sections 37 and 38 from Methods C 26 and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this change may be referred to letter ballot of the Society.[6]

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Definitions of Terms Relating to Gypsum C 11 – 40 T.[3]—In accordance with the recommendation of Committee E-8 on Nomenclature and Definitions, Committee C-11 has revised the definitions of gypsum lath, gypsum sheathing board, and gypsum wall board as follows and asks that these revised definitions be approved for reference to letter ballot of the Society for adoption as standard:

Gypsum Lath.—A sheet or slab having an incombustible core of gypsum, containing not more than 15 per cent by weight of fiber, and surfaced with paper suitable to receive gypsum plaster.

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[5] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 55.

[6] This recommendation was modified at the annual meeting of the Society and action taken to publish the revision for a year as tentative, with the understanding that it would be submitted to letter ballot of the Society for adoption as standard in 1942.

Gypsum Sheathing Board.—A sheet or slab having an incombustible core of gypsum, containing not more than 15 per cent by weight of fiber, and surfaced with a water-resistive paper.

Gypsum Wall Board.—A sheet or slab having an incombustible core of gypsum, containing not more than 15 per cent by weight of fiber, and surfaced with paper suitable to receive decoration.

IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

Standard Specifications for Gypsum (C 22 – 25).[4]—The committee recommends that the revision of Section 7(*b*), including the note following Paragraph (*c*), of Specifications C 22 published as tentative last year[7] be approved for reference to letter ballot of the Society for adoption as standard.

V. EDITORIAL REVISIONS OF STANDARDS

Standard Specifications for Gypsum Wall Board (C 36 – 34),[8] *Gypsum Lath (C 37 – 40),*[9] *and Gypsum Sheathing Board (C 79 – 34).*[10]—In Section 1 of each of these specifications the product is referred to as a "gypsum plaster board." Inasmuch as the term gypsum plaster board is being deleted from the Tentative Definitions of Terms Relating to Gypsum (C 11 – 40 T), the committee recommends an editorial change in each of these specifications. In Specifications C 37 substitute the words "which are" for the words "gypsum plaster boards;" and in Specifications C 36 and C 79 substitute the words "which is" for the words "a gypsum plaster board."

In Section 3 of each of these specifications reference is made to Sections 38 to 40 of the Standard Methods of Testing Gypsum and Gypsum Products (C 26 – 40).[5] The addition or omission of a section in Methods C 26 requires the renumbering of subsequent sections which may make necessary changes in the references to these methods which appear in other standards. The committee therefore recommends the omission of the words "Section 38 to 40 of" from Section 3 of these three specifications.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 23 members; 23 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not voting"
I. REVISION OF TENTATIVE STANDARD			
Definitions of Terms Relating to Gypsum (C 11 – 40 T)....	23	0	0
II. REVISIONS OF STANDARDS			
Spec. for Gypsum (C 22 – 25), immediate adoption........	23	0	0
Spec. for Gypsum Partition Tile or Block (C 52 – 33), immediate adoption........	21	0	2
Methods of Testing Gypsum and Gypsum Products (C 26 – 40)................	22	0	1
III. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Definition of Terms Relating to Gypsum (C 11 – 40 T)....	22	0	1
IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Spec. for Gypsum (C 22 – 25)..	23	0	0

This report has been submitted to letter ballot of the committee which consists of 23 members; 23 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

H. J. SCHWEIM,
Secretary.

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[7] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 337.

[8] 1939 Book of A.S.T.M. Standards, Part II, p. 84.

[9] 1940 Supplement to Book of A.S.T.M. Standards Part II, p. 53.

[10] 1939 Book of A.S.T.M. Standards, Part II, p. 82.

APPENDIX

PROPOSED METHOD OF TEST FOR CALCIUM SULFATE IN GYPSUM AND GYPSUM PRODUCTS BY THE AMMONIUM ACETATE METHOD[1]

This is a proposed method and is published as information only. Comments are solicited and should be addresssed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

1. *Scope.*—This method covers the procedure for determining calcium sulfate in gypsum and gypsum products by extraction with ammonium acetate solution.

2. *Reagents:* (*a*) *Ammonium Acetate Solution.*—Dissolve 454 g. of c.p. ammonium acetate in 2 liters of water. Add sufficient NH_4OH to make the solution distinctly ammoniacal, using phenolphthalein as the indicator.

(*b*) *Ammonium Hydroxide Wash Solution.*—Dilute 100 ml. of NH_4OH (sp. gr. 0.90) to 1 liter.

(*c*) *Filter Aid.*—Analytical grade diatomaceous silica.

3. *Procedure Using Gooch Crucible:* (*a*) Weigh rapidly approximately 4 g. (Note 1) of the well-mixed sample and transfer to a 600-ml. beaker.

NOTE 1.—All weighings shall be made to 0.001 g., except that weights of crucibles and of their contents shall be determined to 0.0001 g.

(*b*) Without delay, weigh 1 g. of the well-mixed sample in a tared weighing bottle having a ground-glass stopper. Dry the sample and weighing bottle to constant weight at 45 C. (Notes 2 and 3), and calculate the percentage loss in weight at 45 C. (largely uncombined water) as follows:

$$\text{Loss in weight at 45 C., per cent} = \frac{A - B}{C} \times 100$$

where:

A = original weight of sample and weighing bottle,

B = weight of sample and weighing bottle dried to constant weight at 45 C., and

C = original weight of sample.

Calculate the weight of the 4-g. sample (Paragraph (*a*)) corrected for loss on heating to constant weight at 45 C.

If the percentage by weight of combined water held by the calcium sulfate is desired, heat the sample and weighing bottle to constant weight at 220 C., and calculate the percentage of combined water as follows:

$$\text{Combined water, per cent} = \frac{B - D}{B - E} \times 100$$

where:

B = weight of sample and weighing bottle dried to constant weight at 45 C.,

D = weight of sample and weighing bottle dried to constant weight at 220 C., and

E = weight of weighing bottle.

NOTE 2.—Avoid overheating in all oven drying of ammonium acetate residues, that is, place crucibles well away from the heating elements. This is of particular importance for samples high in impurities, as these impurities often have water of hydration that is lost on local overheating.

NOTE 3.—Stopper weighing bottles immediately upon removal from the oven in order to prevent absorption of moisture from the air upon cooling.

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[1] This proposed method was prepared by L. S. Wells and W. F. Clarke, National Bureau of Standards, and modified by B. E. Kester, United States Gypsum Co. This proposed method is under the jurisdiction of the A.S.T.M. Committee C-11 on Gypsum. Published as information, June, 1941.

(*c*) To the contents of the 600-ml. beaker (Paragraph (*a*)), add 350 ml. of the ammonium acetate solution, and stir the mixture thoroughly so as to loosen all of the solid matter from the bottom of the beaker. Add 0.2000 g. of redried diatomaceous silica to the mixture, heat the beaker and contents to 70 C. on a steam bath, and maintain at that temperature for 30 min., while stirring frequently. During the heating, keep the solvent ammoniacal by additions of NH_4OH and phenolphthalein, if indicated. (Meanwhile, heat a supply of the ammonium acetate solution to 70 C., keeping it also distinctly ammoniacal.) Filter the mixture, with suction, through a tared Gooch crucible, stirring frequently during filtration to keep the diatomaceous earth suspended in the liquid. Wash the Gooch crucible containing the residue with five 10-ml. portions of the warm acetate solution, draining thoroughly after each washing. Wash in the same manner with eight 10-ml. portions of the NH_4OH wash solution. Care shall be exercised to wash the upper walls of the Gooch crucible. Drain the crucible dry with suction, place in an oven at 70 C., and dry to constant weight. Allow the crucible to cool in a desiccator before weighing.

(*d*) Calculate the percentage of $CaSO_4 \cdot XH_2O$ on the basis of the sample dried to constant weight at 45 C., as follows:

$CaSO_4 \cdot XH_2O$, per cent

$$= \frac{F - (G - H)}{F} \times 100$$

where:

F = weight of sample, corrected for loss on heating to constant weight at 45 C.,

G = weight of dried crucible and contents, and

H = weight of crucible plus diatomaceous silica used as filter aid.

4. *Procedure Using Tared Filter Papers*:[2] (*a*) Dry a quarter-folded, 11-cm. quantitative filter paper overnight at 70 C. in a wide-form, glass-stoppered, 30 by 60-mm. weighing bottle. After drying, cool the weighing bottle and paper in a desiccator, and weigh.

(*b*) Treat the sample exactly as described in Section 3 prior to the filtration. Filter the mixture by gravity through an ordinary 7-cm. glass funnel, stirring frequently during filtration to keep the diatomaceous earth suspended in the liquid. Wash the filter paper and residue with five 10-ml. portions of warm acetate solution, draining thoroughly after each washing. Wash in the same manner with eight 10-ml. portions of the NH_4OH wash solution. After final draining replace the paper and residue in the weighing bottle, and dry at 70 C. to constant weight. Cool the weighing bottle, paper, and residue in a freshly prepared desiccator before weighing; this is essential, due to the hygroscopic character of paper.

(*c*) Calculate the percentage of $CaSO_4 \cdot XH_2O$ as described in Section 3 (*d*), substituting the weight of the weighing bottle and filter paper for the weight of the crucible.

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[2] This procedure is suggested as an alternative method where several samples are to be analyzed at once. It has been found that gravity filtration on six samples will proceed as rapidly as it is possible to handle them.

REPORT OF COMMITTEE C-12

ON

MORTARS FOR UNIT MASONRY

Committee C-12 on Mortars for Unit Masonry has held three meetings during the past year: in Atlantic City, N. J., on June 25, 1940; in Washington, D. C., on December 5, 1940 and on March 26, 1941.

The membership at present totals 35, of whom 14 are classified as producers, 8 as consumers, and 13 as general interest members.

In addition to the subcommittees listed in the 1939 and 1940 annual reports, the following working subcommittees have been established:

Working Subcommittee on Mortar for Reinforced Brick Masonry (H. C. Plummer, chairman), and

Working Subcommittee on Mortar for Concrete Masonry Units (R. B. Crepps, chairman).

The Working Subcommittee on Soundness and Volume Change of Mortar (R. E. Davis, chairman) was discharged, having completed its work of preparing a program of tests.

Although but one specification is being offered for publication as tentative at this time, most of the standing and working subcommittees have been active.

Subcommittee II on Methods of Test (F. O. Anderegg, chairman) has been studying effects of curing (absorbent *versus* nonabsorbent molds) and of shape of test specimen on compressive strength, and the durability of mortar when subjected to frost action. Through the Working Subcommittee on Workability and Plasticity (L. A. Wagner, chairman), comparisons of methods for measuring plasticity are being made. Results have been submitted to the committee by M. O. Withey, W. C. Voss, P. S. Roller, and others. Subcommittee II has recently been reorganized with respect to scope. The formulation of methods of test for use with specifications has been transferred to the jurisdiction of Subcommittee III. Subcommittee II will continue as a research subcommittee.

Subcommittee III on Specifications for Mortar is in process of reorganization both in respect to scope and personnel. The working subcommittees under the jurisdiction of Subcommittee III have been quite active. The working Subcommittee on Mortar for Glass Block (G. M. Rapp, chairman) prepared and submitted as information complete proposed specifications for mortar, but is not offering these specifications as a tentative standard until certain marketing policies of the glass block industry have been clarified. The working Subcommittee on Mortar for Concrete Masonry Units (R. B. Crepps, chairman) has held meetings and is making progress in the preparation of specifications. The Working Subcommittee on Mortar for Reinforced Brick Masonry (H. C. Plummer, chairman) has prepared and submitted proposed Specifications for Mortar for Reinforced Brick Masonry, as appended hereto.[1] These specifications are being recommended for publication as tentative, as indicated later in this report. The proposed specifications have as their background, extensive research,[2] both in the United States and abroad, and observations of field practice on the Pacific Coast where this type of masonry is considerably used.

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[1] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 278.

Subcommittee IV on Specifications for Aggregates for Mortar (Stanton Walker, chairman) has been giving consideration to an extensive revision of the Tentative Specifications for Aggregate for Masonry Mortar (C 144 – 39 T) but has not yet submitted a recommendation.

Subcommittee V on Admixtures for Mortar (W. C. Voss, chairman) had referred to it the request of certain producers that specifications for clay used as an admixture for mortar be prepared, but has not yet submitted a recommendation.

The Joint Working Subcommittee (with Committee C-15 on Manufactured Masonry Units) on Efflorescence has continued its research activities.

NEW TENTATIVE STANDARD

The committee recommends for publication as tentative, new Specifications for Mortar for Reinforced Brick Masonry, as appended hereto.[1] The considerations governing the preparation of these specifications are presented in the section of this report concerning Subcommittee III on Specifications for Mortar.

This recommendation[3] has been submitted to letter ballot of the committee which consists of 35 members; 30 members returned their ballots of whom 19 have voted affirmatively, 5 negatively, and 6 marked their ballots "not voting." The classified vote on this recommendation was as follows: Affirmative: 7 producers, 3 consumers, 9 general interests; negative: 2 consumers, 2 producers, and 1 general interest.

This report has been submitted to letter ballot of the committee which consists of 35 members, 21 members returned their ballots, of whom 18 have voted affirmatively and 3 negatively.

Respectfully submitted on behalf of the committee,

J. W. MCBURNEY,
Chairman.

H. C. PLUMMER,
Secretary.

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[2] See Bibliography appearing in *Brick Engineering*, p. 338, published by Structural Clay Products Inst. (1939); also paper by C. W. Hamann and L. W. Burridge, "Reinforced Brick Work," *The Structural Engineer*, April, 1939, p. 249.

[3] The changes in these specifications, presented at the annual meeting, see Summary of Proceedings, p. 33, were approved by letter ballot of the committee which consists of 35 members; 21 members returned their ballots of whom 15 have voted affirmatively, 1 negatively, and 5 members marked their ballots "not voting."

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE

Committee C-13 on Concrete Pipe held one meeting during the year in Chicago, Ill., on March 22, 1941.

As mentioned in the report last year, Committee C-13 presented to the Society subsequent to the 1940 annual meeting through Committee E-10 on Standards tentative revisions of the Standard Specifications for Concrete Sewer Pipe (C 14 - 35), for Reinforced Concrete Sewer Pipe (C 75 - 35), and for Reinforced Concrete Culvert Pipe (C 76 - 37). Due to the extensive nature of the changes, which were described briefly in the 1940 report, the revisions of these three standards were submitted as separate tentative specifications to supersede when adopted the present standards. These tentative specifications, the titles of which are as follows, were accepted[1] by Committee E-10 on August 28, 1940, and appear in the 1940 Supplement to Book of A.S.T.M. Standards:

Tentative Specifications for:

Concrete Sewer Pipe (C 14 - 40 T),
Reinforced Concrete Sewer Pipe (C 75 - 40 T), and
Reinforced Concrete Culvert Pipe (C 76 - 40 T).

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

As the result of a detailed review of the three tentative specifications at its meeting in Chicago on March 22, the committee now recommends that they be adopted as standard, with minor revisions in the specifications as given below.

These revised specifications will then supersede the present standard specifications.

Tentative Specifications for Concrete Sewer Pipe (C 14 - 40 T),[2] revised as follows:

Section 5.—Change from its present form: namely,

> 5. Portland cement shall conform to the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 9) or the Standard Specifications for High-Early-Strength Portland Cement (A.S.T.M. Designation: C 74), of the American Society for Testing Materials.

to read as follows:

> 5. Portland cement shall conform to the requirements for type I or type III as prescribed in the Standard Specifications for Portland Cement (A.S.T.M. Designation: C 150), of the American Society for Testing Materials.[3]

Tentative Specifications for Reinforced Concrete Sewer Pipe (C 75 - 40 T),[2] revised as follows:

Section 5.—Make the same change in this section as recommended above in Section 5 of Specifications C 14.

New Section.—Add a new Section 15 on Joint Reinforcement to read as follows, renumbering the remaining sections accordingly:

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee C-13 reported the following results of the letter ballot vote of a total of 24 ballots returned from a committee membership of 26: C 14, affirmative 24, negative 0; C 75 and C 76, affirmative 22, negative 2.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 235, 243.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

15. In all pipe 36 in. or more in diameter, the joint shall have a circumferential reinforcement equal in area to that of a single line within the barrel of the pipe.

Section 18.—Renumber as Section 19 and add the following new sentence between the first and second sentences:

The selection shall be made at the point or points designated by the purchaser when placing the order.

Section 19.—Renumber as Section 20 and change from its present form to read as follows:

20. A purchaser of pipe, whose needs require shipments at intervals over extended periods of time, shall be entitled to preliminary tests of not more than three sections of pipe covering each size, 12 to 72 in. in diameter, in which he is interested. The acceptability of 78 to 108-in. pipe shall be determined by tests of the quality of the concrete as placed in the pipe and by examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined from 6 by 12-in. test cylinders made from the concrete used in making the pipe and manufactured and cured under identical conditions with the pipe. Compression tests of such cylinders shall be made in accordance with the Standard Method of Test for Compressive Strength of Concrete (A.S.T.M. Designation: C 39) of the American Society for Testing Materials.[4] After these preliminary tests, a purchaser shall be entitled to additional tests in such numbers and at such times as he may deem necessary, provided that the total number of pipe tested shall not exceed 0.5 per cent of the pipe delivered.

Section 25.—Renumber as Section 26 and change from its present form to read as follows:

26. By agreement between the purchaser and the manufacturer, the acceptability of all sizes of pipe may be determined by tests of the quality of concrete as placed in the pipe and the examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined as specified in Section 20.

Section 29.—Change Paragraph (*b*) from its present form to read as follows:

(*b*) Variations of the position of the reinforcement shall not exceed $\frac{1}{4}$ in. from the position provided in the design for pipe having an internal diameter of 48 in. or less, and shall not exceed $\frac{1}{2}$ in. for larger pipe; but the cover on the reinforcement shall be not less than $\frac{3}{4}$ in. at any point.

Add a new Paragraph (*c*) to read as follows:

(*c*) The underrun in length of pipe from that specified shall be not more than $\frac{1}{8}$ in. per ft. with a maximum of $\frac{1}{2}$ in. in any length of pipe.

Tentative Specifications for Reinforced Concrete Culvert Pipe (*C 76 – 40 T*),[5] revised as follows:

Section 5.—Make the same change in this section as recommended above in Section 5 of Specifications C 14.

Section 11.—Change the second sentence to read as follows by the addition of the italicized words:

In circular pipe having two lines of circular reinforcement, each line shall be so placed that the net protective covering of concrete over the reinforcement *in the barrel of the pipe* shall be not less than 1 in.

Section 15.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

15. [In bell and spigot pipe, and in tongue and groove] *In all* pipe 36 in. or more in diameter, the joint shall have a circumferential reinforcement equal in area to that of a single line within the barrel of the pipe.

Section 20.—Add the following new sentence between the first and second sentences:

The selection shall be made at the point or points designated by the purchaser when placing the order.

Section 21.—Change from its present form to read as follows:

21. A purchaser of pipe, whose needs require shipments at intervals over extended periods of

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[4] 1939 Book of A.S.T.M. Standards, Part II, p. 328.

[5] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 253.

time, shall be entitled to preliminary tests of not more than three sections of pipe covering each size, 12 to 72 in. in diameter, in which he is interested. The acceptability of 78 to 108-in. pipe shall be determined by tests of the quality of the concrete as placed in the pipe and by examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined from 6 by 12-in. test cylinders made from the concrete used in making the pipe and manufactured and cured under identical conditions with the pipe. Compression tests of such cylinders shall be made in accordance with the Standard Method of Test for Compressive Strength of Concrete (A.S.T.M. Designation: C 39) of the American Society for Testing Materials.[3] After these preliminary tests, a purchaser shall be entitled to additional tests in such numbers and at such times as he may deem necessary, provided that the total number of pipe tested shall not exceed 2 per cent of the pipe delivered.

Footnote 8.—Delete the phrase "It is recommended that" from the beginning of this footnote.

Section 28.—Change from its present form to read as follows:

28. By agreement between the purchaser and the manufacturer, the acceptability of all sizes of pipe may be determined by tests of the quality of concrete as placed in the pipe and the examination of the quality, amount, and accuracy of placement of the reinforcement. The strength of the concrete shall be determined as specified in Section 21.

Section 32.—Add a new Paragraph (*c*) to read as follows:

(*c*) The underrun in length of pipe from that specified shall be not more than $\frac{1}{8}$ in. per ft. with a maximum of $\frac{1}{2}$ in. in any length of pipe.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 26 voting members; 26 members returned their ballots, with the results shown Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Concrete Sewer Pipe (C 14 - 40 T), to replace Standard Specifications C 14 - 35	23	2	1
Spec. for Reinforced Concrete Sewer Pipe (C 75 - 40 T), as revised, to replace Standard Specifications C 75 - 35[a]	20	5	1
Spec. for Reinforced Concrete Culvert Pipe (C 76 - 40 T), as revised, to replace Standard Specifications C 76 - 37[b]	20	5	1

[a] The classified vote on the adoption as standard of the Tentative Specifications for Reinforced Concrete Sewer Pipe (C 75 - 40 T) was as follows: Affirmative: 9 producers, 9 consumers, 2 general interests; negative: 1 producer, 3 consumers, 1 general interest.

[b] The classified vote on the adoption as standard of the Tentative Specifications for Reinforced Concrete Culvert Pipe (C 76 - 40 T) was as follows: Affirmative: 9 producers, 9 consumers, 2 general interests; negative: 1 producer, 3 consumers, 1 general interest.

This report has been submitted to letter ballot of the committee which consists of 26 voting members; 26 members returned their ballots, of whom 22 voted affirmatively and 3 negatively.

Respectfully submitted on behalf of the committee,

THEODORE DOLL,
Chairman.

M. W. LOVING,
Secretary.

REPORT OF COMMITTEE C-14
ON
GLASS AND GLASS PRODUCTS

Two meetings have been held this year by Committee C-14 on Glass and Glass Products: the first in Atlantic City, N. J., on June 25 and 26, 1940, and the second at Baltimore on April 1, 1941. A number of subcommittee meetings have also been held.

Membership changes include the addition of two new members, and a change in representation of one member. The present membership is 41, of whom 13 are classified as producers, 15 as consumers, and 13 as general interests members.

I. New Tentative Standard

On the recommendation of Subcommittee I on Nomenclature and Definitions, the committee submits for publication as tentative the Definition of the Term Glass as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee which consists of 41 members; 29 members returned their ballots, of whom 28 have voted affirmatively, 0 negatively, and 1 member marked his ballot "not voting."

II. Tentative Standards Continued as Tentative

The committee recommends that the following methods be continued in the tentative status for another year:

Tentative Methods of:

Chemical Analysis of Glass Sand (C 146 – 39 T),
Hydrostatic Pressure Test on Glass Containers (C 147 – 40 T),
Polariscopic Examination of Glass Containers (C 148 – 40 T),
Thermal Shock Test on Glass Containers (C 149 – 40 T), and
Flexure Testing of Glass (C 158 – 40 T).

Activities of Subcommittees

Subcommittee I on Nomenclature and Definitions (F. C. Flint, chairman).—This subcommittee, after several meetings, has proposed a Definition of Glass which has been submitted to the committee and approved as indicated earlier in this report. The definition is recommended for publication by the Society as tentative.

Subcommittee II on Chemical Analysis. (G. E. F. Lundell, chairman).—Subsequent to the June, 1940, meeting, Subcommittee II completed the preparation of referee and routine methods for the chemical analysis of soda-lime glass.

These were discussed at the fall meeting of the Glass Division of the American Ceramic Society at Hershey, Pa., revised in the light of the comments that were made, and circularized among the members of the committee for final comment.

The methods as finally drawn up were then submitted to five laboratories for trial in the analyses of the National Bureau of Standards Standard Samples Nos. 80 and 128 of soda-lime glass. A report of this work has been made at the 1941 annual meeting of the American Ceramic Society.

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[1] This definition was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 320.

These proposed methods of Chemical Analysis of Soda-Lime Glass have been submitted to Committee C-14 for letter ballot with the recommendation that they be approved for publication as tentative. The methods will be presented to the Society subsequent to the annual meeting through Committee E-10 on Standards for publication as tentative.[2]

Subcommittee III on Chemical Properties (F. W. Adams, chairman).—As a result of several meetings at which pertinent papers were read, new tentative methods are being developed on the subject of chemical durability. The titles of the papers are as follows:

"Effect of Storage Conditions on Weathering and Chemical Durability of Commercial Glass Containers," by J. S. Owens and E. C. Emanuel.

"Factors Influencing Autoclave Chemical Durability Tests of Glass Containers," by A. Herman.

"Investigation of Variable Factors in Accelerated Autoclave Chemical Durability Tests," by J. S. Owens and E. C. Emanuel.

"Method of Testing Surface Alkalinity of Glass Bottles," by A. J. Liebmann and M. Rosenblatt.

Subcommittee IV on Physical and Mechanical Properties (J. T. Littleton, chairman).—The work of several years culminated recently in the completion of the Tentative Method of Flexure Testing of Glass (C 158 - 40 T). The subcommittee is now concerning itself with further work in an endeavor to propose a method for the determination of the thermal endurance of glass. The shape of the test specimen is being given much consideration. Such shapes as blown articles, drawn rods and fibers, plates and marbles are being considered. Several laboratories have been at work on this problem.

Subcommittee V on Thermal Properties (E. W. Tillotson, chairman).—No projects have been submitted to this subcommittee for immediate solution.

Subcommittee VI on Glass Construction Block and Tile (J. P. Staples, chairman).—The subcommittee has been busy with tests on individual glass block, on panels of glass block, and on the mortars used with these blocks. This program has been in progress several years and will lead to some important specifications and methods.

This report has been submitted to letter ballot of the committee which consists of 41 members; 41 members returned their ballots, of whom 30 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. W. MOREY,
Chairman.

L. NAVIAS,
Secretary.

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[2] See Editorial Note below.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-14 presented to the Society through Committee E-10 on Standards new Tentative Methods of Chemical Analysis of Soda-Lime Glass. These methods were accepted by Committee E-10 for publication as tentative on August 25, 1941, and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 300, bearing the A.S.T.M. serial designation C 169 - 41 T.

REPORT OF COMMITTEE C-15

ON

MANUFACTURED MASONRY UNITS

Committee C-15 on Manufactured Masonry Units held three meetings during the year: in Atlantic City, N. J., on June 26, 1940, during the annual meeting of the Society, and in Washington, D. C., on December 6, 1940, and on March 5, 1941.

The activities of Committee C-15 during the past year have been confined largely to the development and revision of specifications for clay products. In response to requests from manufacturers, new specifications for vitrified clay block for trickling filters have been prepared. These products serve as a combination underdrain and ventilating block for the floors of filter beds in the trickling filters of waste treatment plants. The blocks provide structural support for the filtering media and openings for the distribution and passage of air through the media and for drainage of the effluent. In addition, the tentative revisions of the methods for testing brick (C 67 – 39) and of the specifications for structural clay tile (C 34 – 39 and C 56 – 39) are being recommended for adoption as standard.

In the past, the committee as a whole has given much attention to the preparation of provisions which would clarify the requirements for qualities of masonry units that are judged by visual examination. Progress has been slow, but a special working committee under the chairmanship of T. I. Coe has now prepared a draft covering requirements that need to be met in the visual examination of clay building brick which has been accepted in principle and referred to Subcommittee II on Clay Building Brick for consideration and action.

Subsequent to the 1940 annual meeting, Committee C-15 presented to the Society, through Committee E-10 on Standards, the following recommendations, which were accepted[1] by Committee E-10 on August 28, 1940, and appear in the 1940 Supplement to Book of A.S.T.M. Standards:

Revision of Tentative Specifications for:

Paving Brick (C 7 – 38 T),

Tentative Revisions of Standards:

Specifications for Structural Clay Load-Bearing Wall Tile (C 34 – 39),

Specifications for Structural Clay Non-Load-Bearing Tile (C 56 – 39), and

Methods of Sampling and Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 – 39).

Since the 1939 annual meeting, 3 new members have been elected and 2 resignations accepted. The committee membership now totals 58, of whom 13 are classified as consumers, 27 as producers, and 18 as general interest members.

I. NEW TENTATIVE STANDARD

The committee recommends that the proposed Tentative Specifications for Vitrified Clay Filter Block for Trickling Filters[2] be accepted for publication as tentative.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee C-15 reported the following results of the letter ballot vote of a total of 47 ballots returned from a committee membership of 57: C 7 – 38 T, affirmative 39, negative 0, ballots marked "not voting" 8; C 34 – 39 and C 56 – 39, affirmative 45, negative 0, ballots marked "not voting" 2; C 67 – 39, affirmative 44, negative 0, ballots marked "not voting" 3.

[2] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 272.

II. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the tentative revisions of the following standards which were published as tentative last year[3] be approved for reference to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Structural Clay Load-Bearing Wall Tile (C 34 – 39),[4] and

Structural Clay Non-Load-Bearing Tile (C 56 – 39).[4]

Standard Methods of:

Sampling and Testing Brick (C 67 – 39),[5] comprising changes in Sections 7 and 18 and the incorporation of the Tentative Method of Freezing-and-Thawing Test of Brick (C 67 – 39 T).[6]

The recommendations appearing in this report have been referred to letter ballot of the committee which consists of 58 members; 42 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARD			
Specifications for Vitrified Clay Filter Block for Trickling Filters	33	1	8
II. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Specifications for Structural Clay Load-Bearing Wall Tile (C 34 – 39)	41	0	1
Specifications for Structural Clay Non-Load-Bearing Tile (C 56 – 39)	41	0	1
Methods of Sampling and Testing Brick (C 67 – 39)	42	0	0

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Clay Building Brick (J. W. McBurney, chairman) has prepared a draft of a preface or explanatory note intended to indicate the basis for the requirements of the Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 – 40 T),[7] and to inform purchasers when supplementary specifications are desirable.[8] The subcommittee has made further studies of laboratory data on the resistance of brick to freezing and thawing, and has devoted much attention to the difficult problem of classifying bricks having neither an excellent nor a poor record in tests or service.

No unfavorable comment on the tentative revisions[3] of the Standard Methods of Sampling and Testing Brick (C 67 – 39) was received during the year, and the committee is recommending their adoption as standard, as mentioned earlier in this report.

Subcommittee VI on Sewer Brick (T. R. Lawson, chairman) has delayed the preparation of recommendations relating to the Tentative Specifications for Sewer Brick (Made from Clay or Shale) (C 32 – 39 T), pending further consideration of suggestions for minor revisions of the absorption requirements.

Subcommittee VII on Glazed Brick and Tile (J. R. Kauffman, chairman) realizes that the Tentative Specifications for Glazed Building Units (C 126 – 39 T) are incomplete and do not reflect the most recent improvements in the products and testing procedures. However, it has been reluctant to recommend changes pending the receipt of information to be obtained from surveys and investigations.

Subcommittee VIII on Structural Clay Tile (H. C. Plummer, chairman), in response to urgent requests and in the absence of the receipt of unfavorable comment, this subcommittee has approved the adoption as standard of the tentative revisions[3] which clarify the distinction between unscored and facing tile in the Standard Specifications for Structural Clay Load-Bearing Wall Tile (C 34 – 39) and in the Specifications for Structural Clay Non-Load-Bearing Tile (C 57 – 39). The subcommittee has obtained information from manufacturers on reasonable tolerances on dimensions of facing tile and is preparing a draft of a specification for this product.

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 338.

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 108, 112.

[5] *Ibid.*, p. 104.

[6] *Ibid.*, p. 930.

[7] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 180.

[8] See Editorial Note, p. 304.

Subcommittee IX on Clay Filter Block (A. S. Brewer, chairman) was organized during the year to complete the preparation of specifications for filter block. Prior to the appointment of the subcommittee, a special working committee (L. M. Christie, chairman) had prepared a draft of a specification acceptable, in general, to both producers and consumers. The proposed specifications, which are now recommended for publication as tentative, appear to define the product as well as can be with the existing information on physical properties and service performance.

This report has been submitted to letter ballot of the committee which consists of 58 members; 45 members returned their ballots, of whom 44 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. E. PARSONS,
Chairman.

J. W. WHITTEMORE,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-15 presented to the Society through Committee E-10 on Standards a revision of the Tentative Specifications for Building Brick (Made from Clay or Shale) (C 62 – 40 T). The tentative specifications in their revised form were accepted by Committee E-10 on August 25, 1941, and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 267.

REPORT OF COMMITTEE C-16

ON

THERMAL INSULATING MATERIALS

Committee C-16 on Thermal Insulating Materials submits this as its first formal report since its organization in 1938. At a meeting in Rochester, N. Y., on March 10, 1938, preliminary organization was effected, and organization was completed at a meeting held during the 1938 annual meeting in Atlantic City, N. J. Further meetings of the committee and/or its subcommittees were held in Pittsburgh, Pa., on November 21, 1938; in Columbus, Ohio, on March 9, 1939; in Washington, D. C., on October 26, 1939; in Detroit, Mich., on March 7, 1940; in Pittsburgh, Pa., on October 24, 1940; and in Washington, D. C., on March 6, 1941. At the latter meeting, an amendment to the scope of the committee was adopted which extended the scope of the committee to include consideration of all forms of thermal insulating materials except insulation refractories. Action was also taken at that time to increase the membership of the committee to include representation of those especially interested in building insulation. The scope of the committee is now as follows:

Scope.—Formulation of test methods and specifications relating to thermal insulating materials except those used in the insulating refractory fields.

NEW TENTATIVE STANDARDS

The committee recommends that the following proposed methods of test and definitions of terms, as appended hereto,[1] be accepted for publication as tentative:

Methods of:

Test for Compressive Strength and Flexural Strength of Preformed Block Type Thermal Insulating Materials, proposed by Subcommittee II,

Mixing Thermal Insulating Cement, proposed by Subcommittee III,

Test for Bulk Density of Thermal Insulating Cement, proposed by Subcommittee III,

Test for Covering Capacity and Volume Change upon Drying of Thermal Insulating Cement, proposed by Subcommittee III,

Test for Thickness and Density of Blanket Type Thermal Insulating Materials, proposed by Subcommittee IV, and

Definitions of:

Terms Relating to Thermal Insulating Materials, covering "thermal insulating block," and "thermal insulating cement," as proposed by Subcommittees II and III.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 33 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
NEW TENTATIVE STANDARDS			
Test for Compressive Strength and Flexural Strength of Preformed Block Type Thermal Insulating Materials	20	0	2
Method of Mixing Thermal Insulating Cement	25	0	1
Test for Bulk Density of Thermal Insulating Cement	25	1	0
Test for Covering Capacity and Volume Change upon Drying of Thermal Insulating Cement	25	0	1
Test for Thickness and Density of Blanket Type Thermal Insulating Materials	26	1	0
Definitions of Terms Relating to Thermal Insulating Materials:			
Preformed thermal insulating block	21	0	1
Thermal insulating cement	26	0	0

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[1] These methods and definitions were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, pp. 293, 286, 288, 290, 296, 298.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Physical Properties of Preformed Block Insulation (R. H. Heilman, chairman).—This subcommittee has prepared the proposed tentative definition of preformed thermal insulating block and the proposed Tentative Method of Test for Compressive Strength and Flexural Strength of Preformed Thermal Insulating Block referred to earlier in this report. The subcommittee has undertaken a study of methods for determining the effect of prolonged heating on preformed block type insulation. One of these methods involve the use of a Ro-tap sieve shaker. Further work on this subject has been planned and will be reported on at the next meeting.

Subcommittee III on Physical Properties of Insulating Cements (H. H. Rinehart, chairman).—The work of this subcommittee has resulted in the preparation of a proposed tentative definition of thermal insulating cement, and the following three proposed tentative methods, which are now being submitted for publication as tentative:

Methods of:

Method of Mixing Thermal Insulating Cement,

Test for Bulk Density of Thermal Insulating Cement, and

Test for Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement.

A preliminary study has been undertaken on compression-shear testing of thermal insulating cements. A preliminary report has been made of this study and plans have been laid for further work on this subject. The subcommittee has also been discussing methods of testing the effect of heating on the stability of thermal insulating cement.

Subcommittee IV on Physical Properties of Blanket, Flexible, Loose-Fill, and Miscellaneous Types of Insulation (R. E. Cryor, chairman).—This subcommittee has completed the proposed Tentative Method of Test for Thickness and Density of Blanket-Type Thermal Insulating Materials mentioned earlier in this report.

The question of measurement of unfiberized content of rock-wool type insulation is being studied by a subgroup of this subcommittee. A second subgroup has been assigned the problem of studying mechanical stability of wool-type insulation after prolonged heating. The use of the "Ro-tap" sieve shaker was suggested as a means of investigating this characteristic. Several members of the subcommittee agreed to conduct tests using this machine. A third subgroup was assigned the task of making a survey of test methods available to determine (*1*) the corrosive effect on metals of blanket insulation under various moisture conditions, and (*2*) the resistance of the insulating material itself to decomposition under various moisture conditions. At the March, 1941, meeting of the subcommittee reports of the three subgroups were considered. The work on these subjects will be carried forward by the three subgroups.

Subcommittee V on Thermal Properties Other Than Thermal Conductivity of All Forms of Thermal Insulation (E. R. Queer, chairman).—A preliminary report on one method for the determination of specific heat of thermal insulating materials was offered to this subcommittee for study and will be reported on at the next meeting.

Subcommittee VI, the A.S.T.M. Group of the Joint Committee on Thermal Conductivity of all Forms of Insulation (F. C. Houghten, chairman).—This subcommittee which comprises the A.S.T.M. representatives on the Joint Committee held two meetings during the past year. The joint committee is sponsored by the American Society of Heating and Ventilating Engineers, the American Society of Refrigerating Engineers, the National Research Council, and A.S.T.M. At the Pittsburgh meeting in October, 1940, a Proposed Tentative Method for Measurement of Thermal Conductivity of Insulating Materials by Use of the Guarded Hot Plate was submitted to Committee C-16. Because of the fact that values of thermal conductivity obtained for given materials in different laboratories usually do not agree within a range considered by many members of Committee C-16 to be reasonable, it was decided not to submit the proposed method to Committee C-16 for approval until tests have been conducted by several laboratories on samples of several kinds of material using the proposed procedure. If the results of these tests agree within limits thought by the members of Committee C-16 to be acceptable, the proposed method of test will be submitted to the membership for letter ballot. If, on the other hand, the results do not agree within acceptable limits, further study of the proposed method will be made with a view toward reducing to such reasonable limits the results obtainable with the guarded hot plate apparatus built and operated in compliance with the proposed method.

Subcommittee VII on Dimensional Standards for Preformed Insulation (Ray Thomas, chairman).—The chairman has submitted to the subcommittee for study a compilation of data on current sizes of preformed insulation offered by manufacturers. This report will be reviewed and action taken on it at the next meeting of the subcommittee.

Subcommittee on Editing (C. B. Bradley, chairman).—This subcommittee has held no meetings but by correspondence has carried out its work of editing the proposed standards prepared by other subcommittees so that they conform to A.S.T.M. practice.

Subcommittee for Liaison with A.S.T.M. Committee C-8 on Refractories (R. H. Heilman, chairman).—This subcommittee held one meeting during the year for the purpose of effecting an agreement between the two cooperating committees on the matter of definition of preformed block insulation.

This report has been submitted to letter ballot of the committee which consists of 33 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. H. WALKER,
Chairman.

E. T. COPE,
Secretary.

REPORT OF COMMITTEE C-18 ON NATURAL BUILDING STONES

The former Committee C-18 on Natural Building Stones and Slate was discharged in 1940, and the following temporary officers were appointed by the Society to reorganize a new committee to cover the same subjects:

Chairman, W. C. Clark.
Vice-Chairman, T. I. Coe.
Secretary, D. W. Kessler.

A reorganization meeting was held in Atlantic City, N. J., on June 26 and 27, 1940, which was attended by six members of the original committee and five prospective new members or their representatives. Since that time 12 other members have been elected to the committee. Of the total membership of 23, 10 are classified as producers, 7 as consumers, and 6 as general interest members.

At the reorganization meeting, plans were made for various activities of the committee, and the following subcommittees were formed:

Subcommittee I, Advisory,
Subcommittee II on Nomenclature and Definitions,
Subcommittee III on Test Procedures,
Subcommittee IV on Quality Specifications,
Subcommittee V on Surface Finishes,
Subcommittee VI on Stone Setting and Maintenance, and
Subcommittee VII on Cubing.

A number of these subcommittees held meetings during the year at which various specifications were discussed. At a later date a Working Subcommittee on Slate Specifications was appointed.

On November 12, 1940, the committee held a meeting in Washington, D. C., with 17 members present. The temporary officers were elected as permanent officers for the usual term of two years. A proposal was made to change the name of the committee from "Natural Building Stones and Slate" to "Natural Building Stones," since this title seemed more logical and less likely to be misconstrued. This change was approved by unanimous vote of the members present. A corresponding change was made in the scope. Slate is still an important part of the work of the committee, however. Discussions concerning future activities centered largely around the subject of cubing. Since several members expressed the view that a standard method for cubing would not be generally used by the various branches of the industry, it was decided to defer further action on that subject until more complete information was obtained as to the need for such a standard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (L. W. Currier, chairman) held meetings on November 13, 1940, and on March 6 and 18, 1941. At the first meeting, the Tentative Definitions of Terms Relating to Natural Building Stone (C 104 – 31 T),[1] which had been formulated by the former committee, were discussed in detail. It was decided that practically all of these were not sufficiently specific. The subcommittee is accordingly recommending that Tentative Definitions C 104 – 31 T be withdrawn.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 946.

New definitions were formulated for the terms granite, gneiss, black granite, quartzite, limestone, dolomite, marble, and verde antique. A draft of these definitions has been submitted to Committee C-18 for comment and criticism, and it is hoped that they will be ready for presentation to the Society as a tentative standard at an early date.

Subcommittee III on Test Procedures (D. W. Kessler, chairman) held a meeting on November 13, 1940, when it was voted to withdraw the following tentative standards:

Tentative Methods of:[2]

Test for Compressive Strength of Natural Building Stone (C 98 – 30 T),

Sampling Natural Building Stone and Preparation of Sample for Testing (C 101 – 32 T), and

Tension Testing of Natural Building Stone (C 103 – 32 T).

A draft of the new compression test procedure was formulated and sent to all of the members of Committee C-18 for comment and criticism. Various changes have been suggested and a revised copy of the procedure has been circulated for further comment. The returns so far received indicate that this test can be put in satisfactory form for submission as a tentative standard at an early date.[3]

Subcommittee IV on Quality Specifications (W. C. Clark, chairman) held a meeting in Washington, D. C., on November 12, 1940. The important features of specifications for natural building stones were discussed, and it was the concensus that a specification for stone should contain requirements for soundness, strength, and absorptive characteristics. It was not thought necessary to have the specifications include a chemical analysis and weight of the material. A specification for limestone, prepared by the Indiana Limestone Institute, was submitted, and probably will be used as a basis for the formulation of an A.S.T.M. specification for this material. Representatives of the marble, sandstone, and granite industries were requested to submit proposed specifications for consideration. A final draft of the specifications will not be prepared until Subcommittee II on Nomenclature and Definitions has completed its description of the materials under consideration.

The Working Subcommittee on Slate Specifications held a meeting on November 12, 1940, with nine members present. The discussions related to various clauses now used in specifications for roofing slate and to means of arriving at a specification that would be more generally applicable to all slates. The subject of fading was indicated to be a matter of considerable importance, and it was suggested that a specific definition of fading is desirable because the term is loosely used to denote more than one type of color change in the weathering of slate. At this meeting it was decided that the Advisory Committee of Committee C-18 should formulate a questionnaire for submission to the members of the Working Committee on Slate in order to obtain a more complete expression of views on several important specification clauses. This has been done and the replies indicate that a laboratory study to devise a suitable fading test is a necessary step in attempting to draft a specification.

Subcommittee V on Surface Finishes (H. L. Smith, chairman) held a meeting on November 12, 1940, in Washington, D. C., at which discussions were held to determine the possibility of establishing adequate definitions for the various types of surface finishes on marble, limestone, sandstone, granite, and slate. The various producer groups were asked to submit descriptions of all their commercial finishes, and photographs when available. To date data have been supplied for 11 finishes for exterior marble, 4 finishes for interior marble, 19 finishes for sandstone, and 23 finishes for limestone.

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[2] 1939 Book of A.S.T.M. Standards, Part II, pp. 940, 935, 943.

[3] See Editorial Note p. 310.

Subcommittee VI on Stone Setting and Maintenance (J. A. McBeath, chairman) held two meetings in Washington, D. C., on November 13, 1940, and on March 16, 1941. The discussions were mainly for the purpose of obtaining practical information from those members of long experience in setting stone masonry and means of making full use of this information in future construction and in the preparation of specifications for natural building stones. The subjects covered were mortars, plastic calking materials, primers, spacers, dowels, staining, and problems in setting stone steps. A working subcommittee was appointed to study and report on setting and pointing mortars for stone masonry. This subcommittee is composed of Alexander Howie, J. A. McBeath, and Norman Campbell.

WITHDRAWAL OF TENTATIVE STANDARDS

In accordance with the recommendations of Subcommittee II on Nomenclature and Definitions and Subcommittee III on Test Procedures, the committee is recommending the withdrawal of the following tentative standards:

Tentative Methods of:[2]

Test for Compressive Strength of Natural Building Stone (C 98 - 30 T),

Sampling Natural Building Stone and Preparation of Sample for Testing (C 101 - 32 T),

Tension Testing of Natural Building Stone (C 103 - 32 T), and

Tentative Definitions of:[1]

Terms Relating to Natural Building Stones (C 104 - 31 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 23 members; 21 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
WITHDRAWAL OF TENTATIVE STANDARDS			
Test for Compressive Strength of Natural Building Stone (C 98 - 30 T)	19	0	2
Sampling Natural Building Stone and Preparation of Sample for Testing (C 101 - 32 T)	19	0	2
Tension Testing of Natural Building Stone (C 103 - 32 T)	19	0	2
Definitions of Terms Relating to Natural Building Stones (C 104 - 31 T)	20	0	1

This report has been submitted to letter ballot of the committee which consists of 23 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

WALTON C. CLARK,
Chairman.

D. W. KESSLER,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee C-18 presented to the Society through Committee E-10 on Standards a new Tentative Method of Test for Compressive Strength of Natural Building Stone. This method was accepted by Committee E-10 for publication as tentative on October 23, 1941, and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 275, bearing the A.S.T.M. serial designation C 170 - 41 T.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 27, 1940, in Atlantic City, N. J., during the annual meeting of the Society, and on March 5, 1941, in Washington, D. C., during the spring group meetings of the Society.

At the time of the annual meeting, a dinner was held for the purpose of recognizing the thirtieth anniversary of the founding of this standing committee and to honor the original members.

The committee recommends that the Society continue as a member-body in the Inter-Society Color Council during the coming year.

Committee D-1, at the last two meetings, arranged for the presentation of the following papers:

"Pictorial Rusting Standards; Their Development and Value to Our Industry," by J. C. Moore and J. W. Iliff,[1]

"Inert Materials for Admixture with Paint Pigments," by W. R. Fuller,[2]

"Review of the Work on Accelerated Testing of Enamel Finishes," by C. Y. Hopkins,

"Methods for Producing Uniform Films," by A. O. Allen,

"Quantitative Adhesion Measurement of Coatings Before and After Exposure," by R. D. Bonney, and

"A Discussion of Specular Gloss," by R. S. Hunter.

During the past year, a special committee was appointed, under the chairmanship of F. C. Schmutz, to develop plans for taking part in the A.S.T.M. Exhibit of Testing Apparatus to be held at the time of the annual meeting.

E. F. Hickson has been appointed as one of the committee's representatives on Technical Committee X on Conditioning and Weathering, of Committee E-1.

During the year, Committee D-1 has recommended submittal by the Society of 17 standards to the American Standards Association for approval as American Standards. Six of the D-1 standards revised at the last annual meeting had ASA approval and were accordingly submitted in their revised form for reapproval as American standard which has since been granted. At the same time approval as American standard of one standard was withdrawn on the recommendation of Committee D-1.

Since the last annual meeting, 36 new members have been elected, 5 resignations have been accepted, and 5 deaths reported, leaving a total membership of 261.

Of the one adviser and fourteen D-1 subcommittees, two furnished reports of progress and nine submitted detailed reports affecting standards, which have resulted in the committee making the recommendations given below to be acted upon at the annual meeting.

In this report, recommendations affecting standards and tentative standards are noted in brief form below, together with the results of the letter ballot. These recommendations are explained in the reports of the subcommittees directly responsible for them.

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[1] See "Proposed Reference Standards of Rusting of Painted Iron or Steel Surfaces," ASTM BULLETIN, No. 107, December, 1940, p. 25.

[2] Published in ASTM BULLETIN, No. 105, August, 1940, p. 35.

I. NEW TENTATIVE STANDARDS

The committee submits for publication as tentative the following nine new specifications and three methods of test:

Specifications for:

- Oiticica Oil (Permanently Liquid), proposed by Subcommittee II,
- Liquid Paint Driers, proposed by Subcommittee IX,
- Diatomaceous Silica Pigment, proposed by Subcommittee XV,
- Barium Sulfate Pigments, proposed by Subcommittee XV,
- Mica Pigment, proposed by Subcommittee XV,
- Aluminum Silicate Pigment, proposed by Subcommittee XV,
- Magnesium Silicate Pigment, proposed by Subcommittee XV,
- Lead Titanate, proposed by Subcommittee XV, and
- Dibutyphthalate, proposed by Subcommittee XXV.

Methods for:

- Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service, proposed by Subcommittee VII,
- Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces, proposed by Subcommittee VII, and
- Test for Color of Orange Shellac, to be added, when adopted as standard, to the Standard Methods of Sampling and Analysis of Shellac (D 29 - 40), proposed by Subcommittee XIII.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative methods of test and definitions of terms be revised as referred to in the reports of the subcommittees and continued as tentative:

Tentative Methods of:

- Sampling and Testing Aluminum Powder and Paste (D 480 - 38 T), proposed by Subcommittee VIII,
- Testing Liquid Driers (D 564 - 40 T), to include new procedures for determining cobalt and iron, proposed by Subcommittee IX, and
- Test for Specular Gloss of Paint Finishes (D 523 - 39 T), proposed by Subcommittee XVIII.

Tentative Definitions of:

- Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 40 T), proposed by Subcommittee VI.

III. REVISIONS OF STANDARDS

The committee recommends for immediate adoption, revisions in the following standards, and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Specifications for (proposed by Subcommittee XV)*:*

- Zinc Oxide (D 79 - 39; ASA K22-1939),
- Leaded Zinc Oxide (D 80 - 40),
- Basic Carbonate White Lead (D 81 - 38; ASA K23-1938),
- Basic Sulfate White Lead (D 82 - 38),
- Red Lead (D 83 - 39; ASA K24-1939),
- Mineral Iron Oxide (D 84 - 40; ASA K25-1940),
- Ocher (D 85 - 40),
- Lampblack (D 209 - 30; ASA K26-1937),
- Bone Black (D 210 - 30; ASA K36-1937),
- Chrome Yellow (D 211 - 40; ASA K27-1940),
- Pure Chrome Green (D 212 - 40),
- Reduced Chrome Green (D 213 - 40; ASA K28-1940),
- Prussian Blue (D 261 - 40; ASA K29-1940),
- Chrome Oxide Green (D 263 - 40; ASA K37-1940),
- Reduced Para Red (D 264 - 40; ASA K31-1940),
- Aluminum Powder for Paints (Aluminum Bronze Powder) (D 266 - 39),
- Gold Bronze Powder (D 267 - 31),
- Blue Lead; Basic Sulfate (D 405 - 38),
- Aluminum Pigment Paste for Paint (D 474 - 39),
- C. P. Para Red Toner (D 475 - 40),
- Titanium Dioxide Pigments (D 476 - 39; ASA K45-1939),
- Zinc Sulfide Pigments (D 477 - 39),
- C. P. Zinc Yellow (Zinc Chromate) (D 478 - 40), and
- Zinc Dust (Metallic Zinc Powder) (D 520 - 40).

Standard Methods of:

Testing Drying Oils (D 555 – 39), proposed by Subcommittee II.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard, and that the three so indicated be revised as referred to in the report of the subcommittees:

Tentative Specifications for:

Raw Soybean Oil (D 124 – 37 T), revised as proposed by Subcommittee II,
Shellac Varnishes (D 360 – 39 T), proposed by Subcommittee XIII,
Ultramarine Blue (D 262 – 40 T), revised as proposed by Subcommittee XV,
Titanated Lithopone (D 477 – 40 T), to be added to the Standard Specifications for Zinc Sulfide Pigments (D 477 – 39), proposed by Subcommittee XV, and
Carbon Black (D 561 – 40 T), revised as proposed by Subcommittee XV.

Tentative Methods of Test for:

Consistency of Enamel Type Paints (D 562 – 40 T), proposed by Subcommittee VIII,
Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus (D 522 – 39 T), proposed by Subcommittee XXV.

V. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the tentative revisions of the following six standards be approved without change for reference to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Raw Tung Oil (D 12 – 33), proposed by Subcommittee II,
Perilla Oil, Raw or Refined (D 125 – 23), proposed by Subcommittee II, and
Orange Shellac (D 237 – 33), proposed by Subcommittee XIII.

Standard Methods of:

Testing Drying Oils (D 555 – 39), proposed by Subcommittee II,
Chemical Analysis of White Linseed Oil Paints (D 215 – 37), proposed by Subcommittee VIII, and
Sampling and Testing Lacquer Solvents and Diluents (D 268 – 33), by the incorporation of the Tentative Methods of Test for Toluene (Toluol) Dilution Ratio of Lacquer Solvents (D 268 – 39 T), proposed by Subcommittee XXV.

VI. EDITORIAL CHANGES IN STANDARD

Editorial changes are being made in the Standard Methods of Sampling and Analysis of Shellac (D 29 – 40), as proposed by Subcommittee XIII.

These recommendations have been submitted to letter ballot of the committee which consists of 209 voting members; 107 members have returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Underground Pipe Protection (F. N. Speller, adviser).—The American Petroleum Institute's underground pipe coating tests have, after ten years in various soils, been examined and the results published in the November, 1940, issue of the Production Proceedings of the Institute.

Subcommittee II on Drying Oils (Francis Scofield, chairman).—This subcommittee recommends for publication as tentative, new Specifications for Oiticica Oil.[3] This is one of the newer oils for which a demand is developing.

The subcommittee recommends for immediate adoption as standard a revision in Standard Methods of Testing Drying Oils (D 555 – 39),[4] as follows:

New Section.—Add a new Section 23 covering the determination of matter insoluble in chloroform to read as follows:

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[3] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 382.

[4] 1939 Book of A.S.T.M. Standards, Part II, pp. 718, 1136.

23. *Matter Insoluble in Chloroform.*—Dissolve 10 g. of the sample in sufficient U.S.P. chloroform to obtain a fluid solution. Filter through a dried and weighed Gooch crucible, wash with chloroform until the oil is removed, and dry at 105 to 110 C. Cool, weigh, and calculate the percentage of matter insoluble in chloroform.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Oiticica Oil (Permanently Liquid)	52	0	55
Spec. for Liquid Paint Driers	60	1	46
Spec. for Diatomaceous Silica Pigment	65	0	42
Spec. for Barium Sulfate Pigments	64	2	41
Spec. for Mica Pigment	65	0	42
Spec. for Aluminum Silicate Pigment	67	0	40
Spec. for Magnesium Silicate Pigment	66	0	41
Spec. for Lead Titanate	63	0	44
Spec. for Dibutylphthalate	56	0	51
Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service	83	0	24
Method for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces	80	0	27
Test for Color of Orange Shellac (D 29 - 41 T)	47	0	60
II. REVISIONS OF TENTATIVE STANDARDS			
Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 40 T)	86	1	20
Sampling and Testing Aluminum Powder and Paste (D 480 - 38 T)	66	0	41
Testing Liquid Driers (D 564 - 40 T)	58	1	38
Test for Specular Gloss of Paint Finishes (D 523 - 39 T)	71	1	35
III. REVISIONS OF STANDARDS			
Spec. for Leaded Zinc Oxide (D 80 - 40), immediate adoption	65	0	42
Sampling paragraph, affecting 24 pigment specifications: D 79 - 39, D 80 - 40, D 81 - 38, D 82 - 38, D 83 - 39, D 84 - 40, D 85 - 40, D 209 - 30, D 210 - 30, D 211 - 40, D 212 - 40, D 213 - 40, D 261 - 40, D 263 - 40, D 264 - 40, D 266 - 39, D 267 - 31, D 405 - 38, D 474 - 39, D 475 - 40, D 476 - 39, D 477 - 39, D 478 - 40, D 520 - 40, immediate adoption	71	0	36
Testing Drying Oils (D 555 - 39), immediate adoption	67	0	40
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Raw Soybean Oil (D 124 - 37 T), as revised	61	1	45
Spec. for Shellac Varnishes (D 360 - 39 T)	54	0	53
Spec. for Ultramarine Blue (D 262 - 40 T), as revised	62	0	45
Spec. for Titanated Lithopone (D 477 - 40 T), to be added to Standard Spec. D 477	62	0	45
Spec. for Carbon Black (D 561 - 40 T), as revised	65	0	42
Test for Consistency of Enamel Type Paints (D 562 - 40 T)	68	0	39
Test for Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus (D 522 - 39 T)	62	0	45
V. ADOPTION AS STANDARD OF TENTATVE REVISIONS OF STANDARDS			
Spec. for Raw Tung Oil (D 12 - 33)	61	0	46
Spec. for Perilla Oil, Raw or Refined (D 125 - 23)	65	0	42
Spec. for Orange Shellac (D 237 - 33)	53	0	54
Testing Drying Oils (D 555 - 39)	70	0	37
Chemical Analysis of White Linseed Oil Paints (D 215 - 37)	71	0	36
Sampling and Testing Lacquer Solvents and Diluents (D 268 - 33), incorporation of Tentative Methods D 268 - 39 T	64	0	43

The subcommittee recommends the adoption as standard of the Tentative Specifications for Raw Soybean Oil (D 124 - 37 T)[4] with the following revision:

Section 2.—In the table of requirements for properties of raw soybean oil, change the maximum percentage for loss on heating at 105 to 110 C. from "0.2" to read "0.3."

The above revision makes the limit correspond to the specification of the American Soybean Processors Association and is necessitated by the widespread use of solvent extracted oil.

The subcommittee further recommends the adoption as standard of the existing tentative revisions[5] in the Standard Specifications for Perilla Oil, Raw or Refined (D 125 - 23), for Raw Tung Oil (D 12 - 33), and Standard Methods of Testing Drying Oils (D 555 - 39).

At the subcommittee meeting in Washington, D. C., on March 3, a paper was presented by I. M. Colbeth on "Specifications on Dehydrated Castor Oil."[6] In this connection the subcommittee has prepared for publication as information in the ASTM BULLETIN Proposed Specifications for Dehydrated Castor Oil.[6]

Subcommittee VI on Definitions (C. H. Rose, chairman).—This subcommittee recommends that the Tentative Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 40 T) be revised as appended hereto.[7] The revision comprises modifications in the definitions for color, drier, hue, and, tint, and the addition of new definitions for baked finish, baking temperature, forced drying temperature, flatting agent, lightness, five types of resins, and saturation. The revision provides for greater accuracy in the definitions, and the new terms are proposed to satisfy a desire on the part of the industry for adequate definitions of the materials and practices covered.

Subcommittee VII on Accelerated Tests for Protective Coatings (H. A. Nelson, chairman).—This subcommittee recommends for publication as tentative, a new Method for Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service,[8] and a new Method for Evaluating Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces.[9] It has been established that the results of paint tests on metal surfaces are affected by the condition of the metal surface and the thickness of the films that comprise the paint system. For this reason it is desirable to have a method whereby these variables can be minimized when comparative tests of paint systems must be made by different individuals or organizations. It is also recognized that the subcommittees of Committee D-1, as well as other groups, are often confronted with the necessity of evaluating the degree of resistance to rusting obtained with paint systems on iron or steel surfaces. One of the most difficult problems encountered in such cases is obtaining consistent agreement among inspectors as to the degree in which the given paint system has prevented failure. Pictorial standards represent the most practical means for expressing the results in terms that can be understood by all interested individuals.

The subcommittee further recommends that the complete report on accelerated testing of paint coatings, to include results of questionnaire, be published in the ASTM BULLETIN.

Considerable progress is reported by the subcommittee in the work of its subgroups. Group 1 on House Paints (C. H. Rose, chairman) is continuing study of laboratory weathering cycles for determining tint retention. Group 2 on Enamels (C. Y. Hopkins, chairman) has developed a procedure for preparing panels for testing enamels on steel and is now considering preparation of specifications to cover aluminum panels. Group 6 on Metal Protective Finishing Systems (J. C. Moore, chairman) is conducting a study of methods for reproducing corrosion and deterioration of metal protective paints through accelerated means. Group 7 on Questionnaire Covering Accelerated Test Methods (H. G. Arlt, chairman) has concluded its work, which resulted in the recommendation as given above.

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[5] 1939 Book of A.S.T.M. Standards, Part II, pp. 1182, 1184, or 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 345, 346.

[6] ASTM BULLETIN, No. 110, May, 1941, pp. 29, 31.

[7] These revised definitions were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 406.

[8] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 395.

[9] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 396.

Subcommittee VIII on Methods of Analysis of Paint Materials (E. F. Hickson, chairman).—This subcommittee recommends that the Tentative Methods of Sampling and Testing Aluminum Powder and Paste (D 480 – 38 T)[10] be revised as indicated below to provide a test procedure for determining coarse particles in aluminum powder that will give more rapid and more accurate results than the method which is being replaced, and continued as tentative:

Section 6 (a).—Change the test for coarse particles in aluminum powder from its present form to read as follows:

6. (*a*) *Aluminum Powder.*—Fill to a depth of not less than 4 in. two straight-walled containers approximately 12 in. in diameter with mineral spirits and fill a third similar container to a depth of not less than 3 in. with acetone. The mineral spirits used for the test shall conform to the Standard Specifications for Petroleum Spirits (Mineral Spirits) (A.S.T.M. Designation: D 235) and the acetone shall conform to the Standard Specifications for Acetone (A.S.T.M. Designation: D 329) of the American Society for Testing Materials. Weigh 5 g. of the sample, transfer to a 250-ml. beaker, and add in small portions a total of about 150 ml. of mineral spirits, mixing *thoroughly* to a uniform consistency after each addition. Clamp a standard sieve,[a] 8 in. in diameter just above the level of the mineral spirits in the first container and pour the sample on the sieve, using a No. 100 (149-micron) sieve[a] for type A powder and a No. 325 (44-micron) sieve[a] for type B powder. Rinse the beaker with clear mineral spirits and transfer the rinsings to the sieve. Holding the sieve at a slight angle, shake it backward and forward, working the sieve alternately just under and just above the level of the liquid. It is important that the material be thoroughly sieved in each container. After the bulk of the sample has passed through the sieve (time required usually about 2 min.) repeat the procedure in the second container for about 2 min. When it is evident that practically none of the residue on the sieve is passing through it, repeat the procedure in the acetone container for about 2 to 3 min., until nothing more passes through the sieve. Rinse the sides of the sieve with a small stream of acetone, and collect the residue at one side of the sieve. Transfer the residue to a dried and weighed aluminum cup or small evaporating dish with a small stream of acetone, using not less than 15 to 25 ml. Place the aluminum cup or dish in an oven, evaporate to dryness at 105 to 110 C., and continue heating at this temperature for 15 min. (*Caution:* Do not close the door of the oven while evaporating the acetone, as there is danger of explosion or flash of acetone vapor.) Cool the dish, weigh. Calculate the residue retained on the sieve from the increase in weight as follows:

$$\text{Residue retained on sieve, per cent} = \frac{\text{wt. of residue}}{\text{wt. of sample}} \times 100$$

NOTE 1.—The end point of the sieving is easily observed after a few tests have been made. The total time for actual sieving should not be more than 15 min. No brush or other aid shall be used during the sieving. It is necessary that all lumps of pigment be thoroughly dispersed in the mineral spirits while in the beaker before transferring to the sieve.

NOTE 2.—Results tend to increase as the acetone becomes fouled with particles of powder. It is therefore important to filter the acetone regularly.

NOTE 3.—A correction factor shall be applied in the case of sieves used in the test that have not been certified by the National Bureau of Standards. Sieves having a correction factor above 1.2 or below 0.8 should not be used. The correction factor may be calculated as follows: Determine the coarse particles of a sample on a master sieve certified by the National Bureau of Standards following the procedure described in Paragraph (*a*). Sieve the same sample on the unknown sieve by the same procedure. Assuming that the residue retained on the certified sieve is 1.0 per cent, and that the residue on the unknown sieve is 1.2 per cent, then the correction factor is 0.833.

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[a] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials, 1939 Book of A.S.T.M. Standards, Parts II and III.

This subcommittee recommends adoption as standard of the Tentative Method of Test for Consistency of Enamel Type Paints (D 562 – 40 T),[11] and the present tentative revision[12] of the Standard

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[10] 1939 Book of A.S.T.M. Standards, Part II, p. 1130.

[11] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 316.

[12] *Ibid.*, p. 348.

Methods of Chemical Analysis of White Linseed Oil Paints (D 215 – 37).

The subcommittee reports progress in connection with the development of a spatula leafing method for aluminum powder and paste, and on a test for consistency of house paints.

Subcommittee IX on Varnish (W. T. Pearce, chairman).—Large quantities of liquid paint driers are used by railroads, the federal government, paint manufacturers, painters, and many others, and it was felt that the Society should develop specifications for the purchase of this class of materials. After considerable study and investigation, a committee composed of representatives of both users and suppliers prepared the proposed Tentative Specifications for Liquid Paint Driers, appended hereto,[13] which were discussed at two meetings of Subcommittee IX before presentation to Committee D-1.

Standard methods for analysis and test for use in the purchase of liquid paint driers have been badly needed. Last year methods for the determination of lead, manganese, and zinc were published as tentative in the Methods of Testing Liquid Driers (D 564 – 40 T). After two years of investigation an electrolytic method for determining cobalt was developed and approved. Inasmuch as this method requires some special apparatus an alternative gravimetric method of slightly less accuracy was developed. Liquid iron driers are purchased in considerable quantities and in consequence a method for iron was developed. These several procedures have been thoroughly tested and approved by a committee representing both users and suppliers and are now being recommended for inclusion in Methods D 564. The methods in their completely revised form are appended hereto.[14]

The subcommittee reports progress in studying methods for determining the color of dry varnish films, and also methods of analysis of alkyd resin solutions.

Subcommittee XIII on Shellac (W. H. Gardner, chairman).—This subcommittee recommends for publication as tentative, a new Method of Test for Color of Orange Shellac.[15] This new method offers a means of expressing the color of orange shellac in numerical values, thus eliminating the necessity for buyer and seller selecting standard samples for color comparison. The method is the result of a comprehensive study of various methods for expressing color of varnish in numerical values. It is very similar to the Angelo color method which has been employed for a number of years in the sale of certain proprietary brands of shellac. Results obtained in cooperative tests using the proposed method are presented in Tables II and III.

Adoption as standard is recommended for the Tentative Specifications for Shellac Varnishes (D 360 – 39 T),[16] with the following minor editorial changes:

Section 6.—In order to indicate the "cut" of the shellac varnish, change the first column of the table of requirements for body of the several types to read as follows by the addition of the italicized words and figures:

Very light *(3-lb. cut)* body varnish
Light body *(4-lb. cut)* varnish
Medium body *(4.5-lb. cut)* varnish
Heavy body *(5-lb. cut)* varnish
Very heavy body *(6-lb. cut)* varnish
Extra heavy body *(8-lb. cut)* varnish

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[13] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 383.

[14] These revised methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 388.

[15] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 385.

[16] 1939 Book of A.S.T.M. Standards, Part II, p. 1137.

Change the note at the end of this section from its present form: namely,

> NOTE.—The terms "very light," "light," "medium," "heavy," "very heavy," and "extra heavy" correspond approximately to the so-called 3, 4, 4.5, 5, 6, and 8-lb. cuts in the trade.

to read as follows:

> NOTE.—The term "cut" is defined as the number of pounds of dry shellac (containing only that amount of moisture acceptable for proper cutting with the solvent to insure a satisfactory varnish) that were added to 1 gal. of specified solvent in manufacturing the varnish.
>
> Allowance has, therefore, been made for 2 per cent maximum moisture including volatile matter, and 3 per cent insoluble matter in orange shellac; and 6 per cent moisture and volatile matter in bleached shellac, in arriving at a minimum value. Varnishes of the above specified bodies or "cuts" should accordingly conform to the respective minimum requirements specified for nonvolatile matter.

TABLE II.—COLOR NUMBERS OF ORANGE SHELLAC SOLUTIONS OBTAINED IN COOPERATIVE TESTS WITH PROPOSED METHOD.

Sample	New York Produce Exchange Laboratory	National Bureau of Standards	Mac Lac	Brooklyn Polytechnic Institute		Average	Maximum Deviation from Average	Average Deviation from Average
				Oper. 1	Oper. 2			
XL	<10	<10	<10	<10	11	<10	...	...
Superfine	10	10	10	11	10	10.2	0.80	0.3
FO Superfine	12	12	12	12.5	14.5	12.60	1.90	0.8
FO Beta	12	11	11	11	12	11.4	0.60	0.5
Delta	13	13	12	13	12.5	12.7	0.70	0.4
London TN	14	16	16	16	15.5	15.5	1.50	0.6
F O Heart	20	23	24	23.5	24	22.8	2.80	1.2
F O TN	24	27	26	27	26	26.0	2.0	0.8

It is pointed out that considerable confusion has resulted from the insistence of the trade in employing pound cuts in designating shellac varnish, and it is therefore believed the above revision will clarify the situation.

The subcommittee recommends that the present tentative revision[17] of the Standard Specifications for Orange Shellac (D 237 – 33) be adopted as standard.

Minor editorial changes are also recommended in the Standard Methods of Sampling and Analysis of Shellac (D 29 – 40),[18] as follows:

Section 12 (e).—Change the description of the starch solution from its present form to read as follows:

> (*e*) *Starch Solution.*—Make a paste of 0.2 g. of soluble (potato) starch in cold water and pour into 100 ml. of boiling water, boil for 5 min., cool, and bottle.

The subcommittee is now engaged in the preparation of a revision of the existing method for determining drying time and the method for determining matter insoluble in cold alcohol.

Subcommittee XV on Specifications for Pigments Dry and in Oil When Marketed in That Form (C. L. Crockett, chairman).—This subcommittee recommends for publication as tentative, as appended hereto,[19] new Specifications for Diatomaceous Silica Pigment, Barium Sulfate Pigments, Mica Pigment, Aluminum Silicate Pigment, Magnesium Silicate Pigment, and Lead Titanate. These five new specifications covering the extender pigments were prepared as a result of requests from consumer interests which originated with the North Dakota State Laboratory in 1939. Lead titanate has also come into use as a pigment and, as a result of consumer requests, specifications for this pigment are also being offered.

The subcommittee was requested by producer interests to revise the footnote appearing in the table of requirements in the Standard Specifications for Leaded Zinc Oxide (D 80 – 40)[20] as it was believed inadequate, as published, for the purpose intended. The following revision of this standard is accordingly recommended for immediate adoption:

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[17] 1939 Book of A.S.T.M. Standards, Part II, p. 1183 or 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 346.

[18] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 142.

[19] These specifications were accepted as tentative by the Society and appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 372 to 377.

[20] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 130.

Section 2 (a).—Change the footnote following the table of requirements for composition and properties of dry pigment from its present form: namely,

> [a] These specification limits may apply to any other combination of zinc oxide and normal or basic lead sulfate that is agreed upon by the purchaser and the seller, but the allowable minimum and maximum limits for zinc oxide content shall be individually agreed upon for each combination.

to read as follows:

> [a] These specification limits and the permissible variation for zinc oxide content (plus 2 and minus 3 per cent, based on the percentage of zinc oxide claimed) apply to any other combination of zinc oxide and normal or basic lead sulfate that is agreed upon by the purchaser and the seller.

The following revised section on sampling or number of tests is proposed for immediate adoption in the case of the 26 pigment specifications listed below. This proposed revision resulted from numerous requests from consumers, producers, and general interest members, the consensus of opinion being that the sampling paragraph as published in the A.S.T.M. specifications cited, does not adequately take care of present-day conditions.

Number of Tests.—Change this section in each of the 26 specifications to read as follows:

> Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10,000 lb., except that for shipments of less than 10,000 lb. two samples shall be taken. At the option of the purchaser the samples may be tested separately, or after blending in equal quantities the samples from the same production unit to form a composite sample.

TABLE III.—CHANGE OF COLOR NUMBERS OF ORANGE SHELLAC SOLUTIONS WITH TIME.

Sample	Cooperator[a]	Age of Varnish, days									
		0	1	2	3	4	6	7	8	9	10
F O Heart	P.I.B.	23	25	29	..	31	33	..	37	..	..
	N.B.S.	23	26	..	31	32	36	..	36	..	..
	Avg.	23	25.5	29	31	31.5	34.5	..	36.5	..	..
F O Superfine	P.I.B.	12	14	16	..	17	18	..	20	..	..
	N.B.S.	12	14	..	..	15.5	16	..	18	..	..
	Avg.	12	14	16	..	16.25	17	..	19	..	..
F O TN	P.I.B.	27	30	31	..	32	33	..	..	35	..
	N.B.S.	27	29	..	35	35	39	..	..	..	..
	Avg.	27	29.5	31	35	33.5	36	..	..	35	..
F O Beta	P.I.B.	11	12	13	..	14	15	..	..	..	..
	N.B.S.	11	..	..	13	14	15	..	..	..	..
	Avg.	11	12	13	13	14	15	..	..	..	..
Superfine	P.I.B.	11	16	18	..	19	20	..	..	20	..
	N.B.S.	10	11	..	..	13.5	14	..	..	..	..
	Avg.	10.5	13.5	18	..	16.25	17	..	..	20	..
London TN	P.I.B.	16	18	22	..	24	27	..	..	27	..
	N.B.S.	16	18	..	..	24	26	..	..	..	..
	Avg.	16	18	22	..	24	26.5	..	..	27	..
Delta	P.I.B.	13	16	17	..	19	20	..	..	21	..
	N.B.S.	13	..	..	15	16	..	18	..	..	..
	Avg.	13	16	17	15	17.5	20	18	..	21	..
XL	P.I.B.	10	11	13	..	14	15	..	15	..	..
	N.B.S.	10	..	..	10	11	13	..	..	..	15
	Avg.	..	..	..	..	12.5	14	..	..	..	..

[a] P.I.B. = Polytechnic Institute of Brooklyn, Brooklyn, N. Y.
N.B.S. = National Bureau of Standards, Washington, D. C.

The above revision affects the following specifications:

Standard Specifications for:

Zinc Oxide (D 79 - 39),[21]
Leaded Zinc Oxide (D 80 - 40),[22]
Basic Carbonate White Lead (D 81 - 38),[21]
Basic Sulfate White Lead (D 82 - 38),[21]
Red Lead (D 83 - 39),[21]
Mineral Iron Oxide (D 84 - 40),[22]
Ocher (D 85 - 40),[22]
Lampblack (D 209 - 30),[21]
Bone Black (D 210 - 30),[21]
Chrome Yellow (D 211 - 40),[22]
Pure Chrome Green (D 212 - 40),[22]
Reduced Chrome Green (D 213 - 40),[22]
Prussian Blue (D 261 - 40),[22]
Chrome Oxide Green (D 263 - 40),[22]
Reduced Para Red (D 264 - 40),[22]
Aluminum Powder for Paints (Aluminum Bronze Powder) (D 266 - 39),[21]
Gold Bronze Powder (D 267 - 31),[21]
Blue Lead; Basic Sulfate (D 405 - 38),[21]
Aluminum Pigment Paste for Paint (D 474 - 39),[21]
C. P. Para Red Toner (D 475 - 40),[22]
Titanium Dioxide Pigments (D 476 - 39),[21]
Zinc Sulfide Pigments (D 477 - 39),[21]
C. P. Zinc Yellow (Zinc Chromate) (D 478 - 40),[22]
Zinc Dust (Metallic Zinc Powder) (D 520 - 40),[22]

Tentative Specifications for:

Ultramarine Blue (D 262 - 40 T),[22] and
Carbon Black (D 561 - 40 T).[22]

The subcommittee recommends for adoption as standard the Tentative Specifications for [23] Titanated Lithopone (D 477 - 40 T) to be added to the Standard Specifications for Zinc Sulfide Pigments (D 477 - 39), Tentative Specifications for Ultramarine Blue (D 262 - 40 T), as revised, and for Carbon Black (D 561 - 40 T), as revised. The revisions in the two latter specifications refer to the new sampling paragraph.

Subcommittee XVIII on Physical Properties of Materials (M. Rea Paul, chairman).—This subcommittee recommends that the present Tentative Method of Test for Specular Gloss of Paint Finishes (D 523 - 39 T) be revised as appended hereto[24] and continued as tentative. This extensive revision of the method represents an attempt to provide a test procedure whereby specular gloss can be more precisely measured.

The subcommittee reports progress on its work of drafting a tentative standard involving use of the I.C.I. method and data in the specification of color; on the development of a standard method of panel preparation for use in specular gloss determinations; and in its work of developing test methods for the adhesion of paint and varnish films and for preparation of uniform paint films.

Subcommittee XXV on Cellulose Ester Coatings (R. M. Carter, chairman).—This subcommittee recommends for publication as tentative, new Specifications for Dibutylphthalate.[25] Dibutylphthalate is a commonly used lacquer plasticizer and is one of the raw materials employed in cellulose ester coatings, specifications for which will generally benefit the lacquer industry.

Adoption as standard is recommended for the Tentative Method of Test for Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus (D 522 - 39 T).[26]

The subcommittee further recommends that the Tentative Method of Test for Toluene (Toluol) Dilution Ratio of Lacquer Solvents (D 268 - 39 T)[27] which represents a tentative revision of the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 33) be adopted as standard.

Subcommittee XXIX on Preparation and Painting of Structural Iron and Steel (A. J. Eickhoff, chairman).—This subcommittee in its report appended hereto presents the results of several field inspections of its pretreated and painted test panels after an exposure of over 21 months.

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[21] 1939 Book of A.S.T.M. Standards, Part II.

[22] 1940 Supplement to Book of A.S.T.M. Standards, Part II.

[23] 1940 Supplement to Book of A.S.T.M. Standards, Part II, pp. 310, 308, 307.

[24] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 401.

[25] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 394.

[26] 1939 Book of A.S.T.M. Standards, Part II, p. 1150.

[27] *Ibid.*, p. 1147.

This report has been submitted to letter ballot of the committee which consists of 209 voting members; 107 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. E. Smith,
Chairman.

M. Rea Paul,
Secretary.

REPORT OF SUBCOMMITTEE XXIX ON PREPARATION AND PAINTING OF STRUCTURAL IRON AND STEEL

Authorized on April 15, 1937, Subcommittee XXIX on Preparation and Painting of Structural Iron and Steel, of Committee D-1, held its organization meeting at Atlantic City, N. J., on June 30, 1938. Due to many diverse opinions concerning the program outlined at this meeting, a Steering Committee was appointed composed of H. E. Smith, chairman of Committee D-1; J. C. Moore, Sinclair Refining Co.; John Pelly Bethlehem Steel Co.; and A. J. Eickhoff, chairman of Subcommittee XXIX. The Steering Committee planned a series of panel tests which it believed would demonstrate the relative merits of several methods of surface preparation (chemical and mechanical).

Within the past few years there has appeared on the market a number of aqueous inhibitive solutions to be applied at atmospheric temperature. These solutions consist essentially of a mixture of water, chromates, reduced chromates, orthophosphoric acid, wetting agents, and metallic phosphates.

This has given rise to the discussion concerning the relative advantages of painting over "cold-pretreated" surfaces, mill scale, sandblasted steel, and surfaces which have rusted, been wire-brushed, and given a cold pretreatment. It is generally recognized that the "hot" type (180 to 200 F.) phosphate-phosphoric acid pretreatment (used extensively on automobile bodies) is definitely advantageous for increasing the paint adhesion and retarding the spread of corrosion between the paint film and the steel surface. However, the "hot" pretreatment is not practical, from the standpoint of field application, for structural steel.

For the present this subcommittee is not concerned with the advantages of different paint formulations. A considerable amount of such work has been done by former A.S.T.M. subcommittees and presented in earlier reports, as follows:

Reports on Inspection of the Havre de Grace Bridge, *Proceedings*, Am. Soc. Testing Mats., Vols. VI to XI (1906–1911); Vol. XIII, p. 332 (1913); Vol. XIV, Part I, p. 232 (1914); Vol. XV, Part I, p. 189 (1915).

Reports on Atlantic City Steel Plate Paint Tests, *Proceedings*, Am. Soc. Testing Mats., Vols. VIII to XI (1908–1911); Vol. XIII, p. 369 (1913); Vol. XIV, Part I, p. 259 (1914); Vol. XV, Part I, p. 214 (1915).

Reports of Subcommittee XIV on Preparation of Iron and Steel Surfaces for Painting, of Committee D-1, *Proceedings*, Am. Soc. Testing Mats., Vols. XVI to XIX, Part I (1916–1919); Vols. 21 to 26, Part I (1921 to 1926); Vol. 28, Part I (1928).

Preparation of Test Panels:

The steel panels used in this test were 2 by 3 ft. by $\frac{3}{16}$ in. hot rolled, copper content not more than 0.06 per cent. The panels to be painted included the following surfaces:

Surface 1.—Original panel with mill scale. Grease and dirt removed with volatile petroleum solvent. Immediately before painting, a large X was scratched diagonally across the panel so as definitely to score the mill scale.

There was some question as to how much protection unbroken mill scale would afford.

Surface 2.—A badly rusted steel panel which was wire brushed before any paint or chemical pretreatment was applied.

Surface 3.—An original panel sandblasted to remove all of the scale. Mr. J. W. Iliff of the E. I. du Pont de Nemours and Co. furnished a special grit to be used in sandblasting. These panels were all sandblasted at Palmerton, Pa., by the New Jersey Zinc Co. One half the number of sandblasted panels were sent to Mr. Rose at the National Lead Co., Brooklyn, N. Y. Forty-eight hours elapsed between the sandblasting and pretreatment or painting.

All panels were backed with a suitable metal protective paint and appropriately marked or numbered. Two locations were selected, namely, Bethlehem, Pa. (an industrial atmosphere) and Sayville, Long Island, N. Y. (a seacoast atmosphere). There were 24 panels at each location. The panels were exposed at an angle of 45 deg. facing south.

Inasmuch as this series of test panels was not designed to study various paint formulations, no description of the paints used is given in this report. Henceforth the paints will be known as primer paint A, finish paint A, and primer paint B, finish paint B. The set of panels treated and painted at Palmerton, Pa., under the direction of G. W. Ashman of the New Jersey Zinc Co. was exposed at Bethlehem, Pa. These panels were numbered 1B, 2B, 3B, etc. The following is the preparation schedule given these panels in carrying out the test procedure suggested by the subcommittee:

July 26, 1939, sixteen panels were sandblasted

July 28, 1939, all of the panels, treated, photographed and prime painted

July 30, 1939, panels second coated

The temperature and relative humidity ranges and the respective average values for each 12-hr. period as based on hourly readings, were as follows:

	Temperature, deg. Fahr., 10 a.m. to 10 p.m.		
	Max.	Min.	Avg.
July 28, 1939	85	75	81
July 30, 1939	82	73	78
	Relative Humidity, per cent 10 a.m. to 10 p.m.		
	Max.	Min.	Avg.
July 28, 1939	96	56	80
July 30, 1939	96	64	78

On August 15, 1939, a small x (about 2 in.) was cut through the paint film to the metal. Panels were crated and sent to Bethlehem, Pa., and put out for exposure (45 deg. facing south) by E. B. Mancke of the Bethlehem Steel Co.

The set of panels exposed at Sayville, Long Island, was prepared at Sayville under the direction of Carlton Rose of the National Lead Co. These panels were numbered 1S, 2S, 3S, etc. The following is the preparation schedule used by Mr. Rose: Panels numbers 1S, 2S, 3S, 4S, 7S, 8S, 9S, 10S, 13S, 14S, 15S, 16S, 19S, 20S, 21S, and 22S were primed July 27, 1939, at a temperature of 81 F., and a relative humidity of 79 per cent. The finish paints were applied on these panels July 31, 1939, temperature 87 F., relative humidity 64 per cent. Panel numbers 5S, 6S, 11S, 12S, 17S, 18S, 23S, and 24S (sandblasted panels) were primed July 28, 1939, temperature 81 F., relative humidity 79 per cent. The finish paints were applied July 31, 1939, temperature 87 F., relative humidity 64 per cent.

Pretreatments of Test Panels:

In reporting the results of the aqueous inhibitors or pretreatments they will be noted as pretreatment *x*, *y*, or *z*, rather than by the individual brand names.

Pretreatment "x" consisted of chromic acid, phosphoric acid, a wetting agent, and some zinc dissolved in the phosphoric acid.

It was used as received (without dilution) and applied by brush. A uniform coat was applied so that the surface was wet evenly without the accumulation of material in recesses or along the bottom edges. The temperature of the solution at the time of application was 80 F. After allowing the film to become thoroughly dry (1 hr.), the surface was ready for a first paint coat. The treated metal surface was not rinsed before applying the paint.

Pretreatment "y" consisted of a saturated solution of zinc phosphate in the minimum amount of phosphoric acid which will keep it in solution.

It was used as received (without dilution) and applied by brush in as heavy a coating as would hold to a vertical surface. The temperature of the solution at the time of application was 80 F. The pretreatment was allowed to air dry on the steel for 4 hr. Then the light-colored coating which had formed on the metal was thoroughly rinsed with clean water. The rinsing was carried out by flushing water on the coated surface with a hose. The rinsed surface was then allowed to air dry thoroughly. After it had dried 1 hr. it received the first coat of paint.

Pretreatment "z" consisted of a homogeneous mixture of phosphoric acid, acid-chromic phosphate, water-soluble oil solvent, wetting agent, and water. This pretreatment was reduced with 3 volumes of hot water (170 to 180 F.) to one volume of pretreatment "z" before application and then applied by brush in sufficient quantity merely to wet the surface. The surface was permitted to become thoroughly and completely dry (2 hr. drying time) before the priming coat of paint was applied. The grayish deposit from the chemical pretreatment was allowed to remain on the panels. The temperature of the solution when applied was approximately 130 F.

Painting of Test Panels:

In order to make the exposures at the two locations as comparable as possible, the spreading rates of the paints were carefully noted.

The spreading rates of paints A and B which were applied to the panels exposed at Bethlehem are shown in the following table:

Panel No. at Bethlehem, Pa.	Primer Paint A, sq. ft, per gal.	Finish Paint A, sq. ft, per gal.
1B	800	600
3B	800	535
5B	800	580
7B	800	605
9B	650	570
11B	695	585
13B	820	600
15B	800	590
17B	710	600
19B	820	605
21B	800	595
23B	780	600

Panel No. at Bethlehem, Pa.	Primer Paint B, sq. ft, per gal.	Finish Paint B, sq. ft. per gal.
2B	775	735
4B	650	710
6B	775	730
8B	790	735
10B	775	710
12B	775	735
14B	775	730
16B	675	700
18B	630	730
20B	775	735
22B	725	710
24B	725	730

The spreading rates of paints A and B applied to the panels exposed at Sayville are shown in the following table:

Panel No. at Sayville, L. I.	Primer Paint A, sq. ft, per gal.	Finish Paint A, sq. ft, per gal.
1S	962	998
3S	934	946
5S	883	1027
7S	815	1027
9S	757	998
11S	935	998
13S	722	946
15S	739	998
17S	756	1027
19S	962	1027
21S	883	1027
23S	794	1027

Panel No. at Sayville, L. I.	Primer Paint B, sq. ft, per gal.	Finish Paint B, sq. ft, per gal.
2S	1015	1127
4S	831	1217
6S	871	1217
8S	1015	1217
10S	926	1127
12S	795	1217
14S	746	1171
16S	732	1217
18S	831	1217
20S	914	1171
22S	892	1171
24S	892	1171

The above tabulations show considerable variation in the spreading rates as applied to the panels at the two painting stations. However, the agreement and uniformity of the spreading rates at a given painting station indicate that the panels have a fairly uniform coat of paint.

Table I gives a summary of the surface conditions of the panels, pretreatments, and surface markings. The letters "B" and "S" (Bethlehem and Sayville) are omitted from the panel numbers. Other than the geographical location and spreading rates, panels 1B and 1S, 2B and 2S, etc., have the same surface, pretreatment, etc.

Prior to the painting and after the final coat of paint had been applied, photographs were taken in order that a definite record of the initial condition of the panels would be available. These pictures include black and white photographs taken by Mr. Ashman and Mr. Rose and Kodachrome photographs taken by Mr. Iliff. At definite intervals Mr. Iliff plans to take further photographs in color of the weathered panels.

The panels at Bethlehem, Pa., and Sayville, Long Island, were put out for exposure on August 15, 1939, at an angle of 45 deg. facing south.

TABLE I.—SUMMARY OF SURFACE CONDITIONS, PRETREATMENTS, AND SURFACE MARKINGS OF TEST PANELS.

Panel No.	Surface Condition	Wire-brushed	Pretreatment	Primer Paint	Finish Paint	Surface Mark
1	Mill scale	No	none	A	A	X
2	Mill scale	No	none	B	B	X
3	Rusted	Yes	none	A	A	..
4	Rusted	Yes	none	B	B	..
5	Sandblasted	No	none	A	A	..
6	Sandblasted	No	none	B	B	..
7	Mill scale	No	x	A	A	X
8	Mill scale	No	x	B	B	X
9	Rusted	Yes	x	A	A	..
10	Rusted	Yes	x	B	B	..
11	Sandblasted	No	x	A	A	..
12	Sandblasted	No	x	B	B	..
13	Mill scale	No	y	A	A	X
14	Mill scale	No	y	B	B	X
15	Rusted	Yes	y	A	A	..
16	Rusted	Yes	y	B	B	..
17	Sandblasted	No	y	A	A	..
18	Sandblasted	No	y	B	B	..
19	Mill scale	No	z	A	A	X
20	Mill scale	No	z	B	B	X
21	Rusted	Yes	z	A	A	..
22	Rusted	Yes	z	B	B	..
23	Sandblasted	No	z	A	A	..
4	Sandblasted	No	z	B	B	..

Inspections of Test Panels:

The test panels at Sayville were inspected by the subcommittee on December 14, 1939 and those at Bethlehem on December 15, 1939. A second inspection of the panels at these two locations was made by the subcommittee on May 28 and 29, 1940, respectively At the May, 1940, inspection it was necessary to sponge lightly with water the test panels at Bethlehem in order to remove the accumulation of dirt, ashes and soot. No cleansing was necessary of the test panels at Sayville. At both of these inspections the panels were rated and an estimate was made of the amount of creepage under the paint film of the small x originally cut in the upper right hand corner of the panel. The results of these inspections are given in Table II.

TABLE II.—REPORT ON INSPECTIONS OF TEST PANELS, DECEMBER, 1939 AND MAY, 1940.

Panel No.	Ratings of Panels at Sayville, L. I., Based on Swedish Pictorial Rusting Standards		Creepage Under Paint Film of Sayville Panels Where Small x was Cut in Upper Right Hand Corner, in.		Ratings of Panels at Bethlehem, Pa., Based on Swedish Pictorial Rusting Standards		Creepage Under Paint Film of Bethlehem Panels Where Small x was Cut in Upper Right Hand Corner, in.	
	Inspection on December 14, 1939	Inspection on May 28, 1940	Inspection on December 14, 1939	Inspection on May 28, 1940	Inspection on December 15, 1939	Inspection on May 29, 1940	Inspection on December 15, 1939	Inspection on May 29, 1940
1	10	10[e]	<1/32	1/16	10	10[j]	1/32	1/8
2	10	10	1/16	1/8	10	10	1/32	1/16
3	7	5	1/32	1/16	10	6	1/32	1/16
4	10	6[f]	<1/32	<1/32	10	9[k]	1/32	1/16
5	10[a]	10[g]	1/32	1/32	10	10	<1/32	1/32
6	10	10	1/32	1/32	10	10	<1/32	1/32
7	10	9	1/16	1/16	10	10	1/16	1/8
8	10	10	1/32	1/32	10	10	1/32	1/16
9	6	3	1/32	1/16	10	7	1/32	1/8
10	10	7[f]	<1/32	1/32	10	9[k]	1/32	1/32
11	10	9	1/32	1/16	10	8	1/32	1/16
12	10	10	1/32	1/32	10	10	<1/32	1/32
13	10[b]	10[h]	1/32	1/16	10	9	1/32	1/16
14	10	10	1/32	1/32	10	10	1/32	1/16
15	9	7	1/16	1/16	10	7	1/16	1/16
16	10	9	1/32	1/32	10	9[k]	1/32	1/16
17	10[c]	9	<1/32	1/16	10	9	1/32	1/32
18	10	10	1/32	1/32	10	10	1/32	1/32
19	10	10	1/16	1/16	10	9	1/32	1/16
20	10[d]	10	1/16	1/16	10	10	1/32	1/8
21	7	4	1/16	1/8	10[i]	5	1/32	1/16
22	10	3[f]	1/32	1/16	10	9[k]	1/32	1/16
23	10	9	1/32	1/16	10	9	1/32	1/32
24	10	10	<1/32	1/32	10	10	<1/32	1/32

[a] With 6 or 7 pin point rust spots.
[b] With 6 rust spots in long X scratch.
[c] With 30 to 35 pin point rust spots.
[d] With a small imperfection in upper left hand corner.
[e] With 8 rust spots.
[f] With subsurface corrosion.
[g] With 25 rust spots.
[h] With continued rusting in diagonal scratch.
[i] With a few incipient breaks in paint film.
[j] With 5 rust spots.
[k] With minute unbroken pin point blisters.

Rather than try to rate the panels as excellent, good, etc., the members of the inspection committee agreed, for the present, to use the Swedish Pictorial Rusting Standards. These standards are colored photographs of painted steel panels which show various degrees of rusting, No. 10 is free from rust, No. 9 shows slight rust, etc., and so on to picture No. 1 which shows a totally rusted surface.

These inspections were made only for the purpose of noting the absence or degree of rusting on the panels. The gloss, dirt retention, chalking, etc., of the paint film was not given any weight or consideration in judging. The object of this study is to determine the amount of protection (free from rust), afforded by the presence or absence of mechanical or chemical treatment of the steel surface prior to painting.

Differences in the degree of rusting of the same type of surface at the two locations may be ascribed to the following:

1. Differences in spreading rates of the paints. On the whole the panels at Bethlehem have a thicker coating of paint than the panels at Sayville.
2. More moisture at Sayville than at Bethlehem.
3. Extra protection is afforded the panels at Bethlehem (due to soot and dirt) which is not afforded the panels at Sayville.

Two further inspections were made of the test panels. Those at Sayville were examined on November 20, 1940, and June 17, 1941, and at Bethlehem on January 21, 1941, and June 18, 1941. The results of these inspections are given in Table III. At these inspections no attempt was made to estimate creepage under the paint film. Instead Mr. Iliff took natural color photographs of this area. Color photographs were also taken of the entire panel.

TABLE III.—REPORT OF INSPECTIONS OF TEST PANELS, NOVEMBER, 1940, AND JANUARY AND JUNE, 1941.

Panel No.	Ratings Based on Swedish Pictorial Rusting Standards			
	Inspection of Panels at Sayville, L. I., November 20, 1940	Inspection of Panels at Bethlehem, Pa., January 21, 1941	Inspection of Panels at Sayville, L. I., June 17, 1941	Inspection of Panels at Bethlehem, Pa., June 18, 1941
1	9[a]	9	7	7
2	10	10	10	10
3	4	5	4	4
4	5[b]	10	5	9
5	8	10	8	10
6	10	10	10	10
7	8	10	8	8
8	10[c]	10	9[d]	10
9	2	6	2	4
10	6	10	5	8
11	7	9	8	9
12	10	10	10	10
13	8	10	8	8
14	10	10	9	10
15	5	6	5	5
16	8[b]	9	8[d]	7
17	7	10	7	10
18	10	10	10	10
19	9	10	9	9
20	9	10	9	9
21	4	5	4	4
22	3	9	3	7
23	8	10	8	10
24	10	10	10	10

[a] With failure along large diagonal X.
With subsurface rusting.
[c] With scattered rust spots in large diagonal X.
[d] With subsurface corrosion.

In Figs. 1, 2, 3, and 4 there is plotted the time of exposure of the test panels in months against the ratings of the panels based on the Swedish Pictorial Rusting Standards, this data being obtained from Tables II and III. Certain portions of the conclusions and observations may be ascertained from a study of these four figures. Other portions of the observations and conclusions are obvious only from a study of the Kodachrome slides.

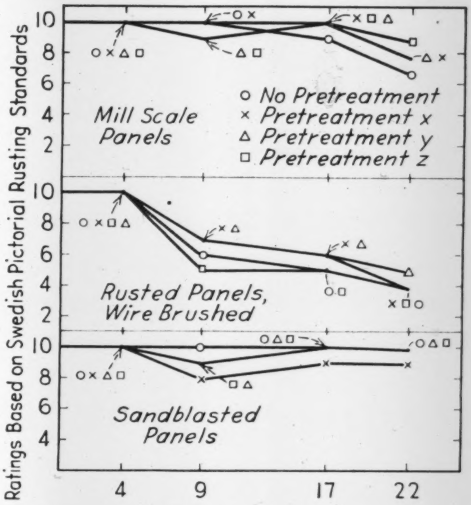

FIG. 1.—Ratings of Paint A on Panels Exposed at Bethlehem, Pa.

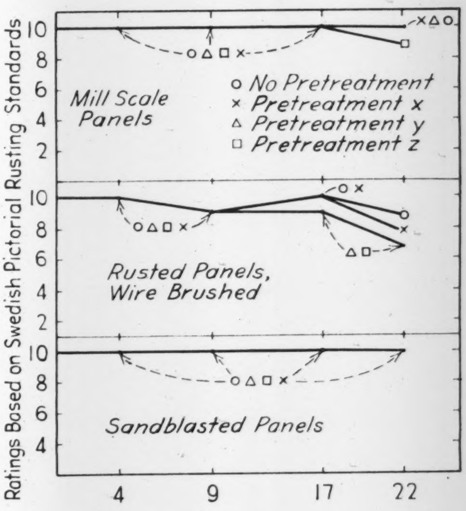

FIG. 2.—Ratings of Paint B on Panels Exposed at Bethlehem, Pa.

Conclusions after 22 Months Exposure:

Now that the panels have been exposed outdoors at 45 deg. south for 22 months, the following conclusions appear to be valid. These conclusions were based only on significant differences in panel performance and a close study of the Kodachrome slides. A significant difference was arbitrarily defined as that which exceeded 2 points on the Swedish Pictorial Rating Scale.

1. Both the "as rolled" and sandblasted panels were comparatively easy surfaces to protect against corrosion by weathering.

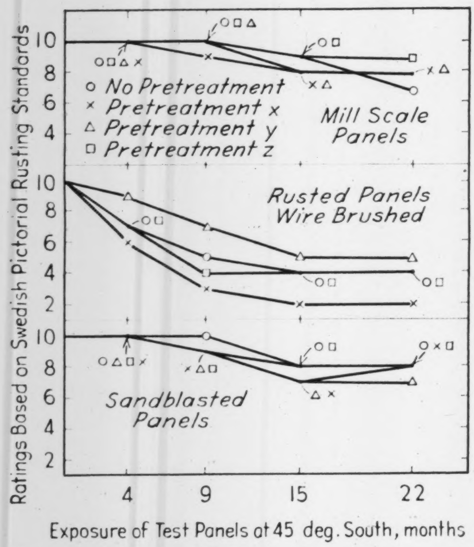

FIG. 3.—Ratings of Paint A on Panels Exposed at Sayville, Long Island, N. Y.

2. The rusted mill scale panels were consistently difficult to protect against corrosion by weathering.

3. Rust creepage at the small x cut on the upper right hand corner was greatest with the "as rolled" steel. This creepage was much less with the rusted steel and practically nil on the sandblasted steel. This was essentially true for all surface treatments (chemical and mechanical).

4. To date none of the chemical treatments have afforded any consistent advantage or disadvantage when applied to iron surfaces under test.

Observations:

The following observations may be of interest:

1. Over the mill scale and sandblasted panels some of the pretreatments are showing slight advantages with paint A. It is therefore possible that these advantages may be extended to paint B when it (paint B) has failed to a considerably greater extent.

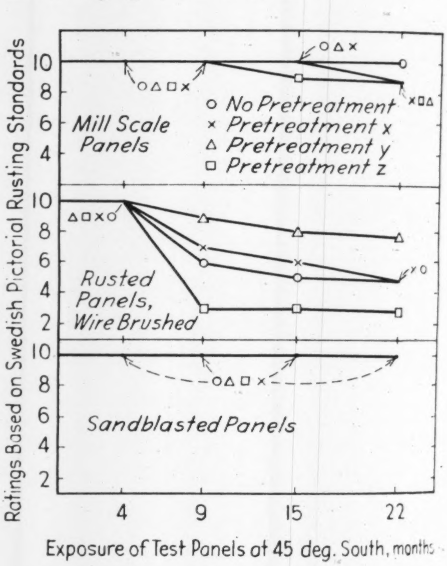

FIG. 4.—Ratings of Paint B on Panels Exposed at Sayville, Long Island, N. Y.

2. It has been noted that the failure of the rusted panels was inconsistent, for example:

(*a*) Slight advantages have been shown for pretreatment *x* under paint A at Bethlehem; slight disadvantages under paint B at Bethlehem and Sayville.

(*b*) Slight advantages were observed for pretreatment *y* under paints A and B at Sayville; slight disadvantages under paint B at Bethlehem.

(*c*) No disadvantage was observed for pretreatment *z* under paint A at Bethlehem or Sayville. Slight disadvantages were observed under paint B at Bethlehem and Sayville.

(*d*) A study of the photographs of the original rusted panels and progress photographs of the paint failures on the panels shows no similarity in pattern.

(*e*) The chemical treatments seem to reduce rust creepage at the small x cut in the upper right-hand corner of the panel. However, this tendency is not consistent.

Acknowledgments:

Acknowledgment is made to the following for their unselfish cooperation and assistance in furnishing labor and materials used in the test:

G. W. Ashman, New Jersey Zinc Co.
V. M. Darsey, Parker Rust Proof Co.
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J. L. Gregg, Bethlehem Steel Co.
R. L. Hallett, National Lead Co.
J. W. Iliff, E. I. du Pont de Nemours and Co., Inc.
C. A. Lominska, National Lead Co.
E. B. Mancke, Bethlehem Steel Co.
H. A. Nelson, New Jersey Zinc Co.
John Pelly, Bethlehem Steel Co.
Carlton Rose, National Lead Co.
F. P. Spruance, American Chemical Paint Co.
J. V. Thompson, Thompson and Co.

Respectfully submitted on behalf of the subcommittee,

A. J. EICKHOFF,
Chairman.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held three regular meetings during the past year (in June at Atlantic City, N. J., in January at Detroit, Mich., and in March at Washington, D. C.).

During the year, Section III on Oxidation of Turbine Oils has been organized in Technical Committee C on Turbine Oils, and Subcommittee XXVII on Classification of Automotive Lubricants and the Special Subcommittee on Federal Specifications Board Methods not A.S.T.M. Standards have been discontinued.

Table I shows the changes in status during the year of American standards and American tentative standards for petroleum products, approved by the American Standards Association on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants.

The voting membership of Committee D-2 now consists of 38 producers, 29 consumers, and 12 general interest members.

Subcommittee V on Viscosity (J. C. Geniesse, chairman), has made a comparison of kinematic and Saybolt Furol viscosities. Seven laboratories determined the Saybolt Furol viscosity at 122 F. on seven samples of oil, and four laboratories determined the kinematic viscosity on the same samples at the same temperature, with results as shown in Table II. The last column of the table contains Saybolt Furol viscosities computed from kinematic viscosities by means of equations developed by W. H. Herschel.

TABLE I.

Title	Current ASTM Designation	Current ASA Number
REVISIONS OF AMERICAN STANDARDS		
Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products	D 86 - 40	Z11.10-194
Test for Distillation of Natural Gasoline	D 216 - 40	Z11.11-1940
Test for Flash Point by Means of the Pensky-Martens Closed Tester	D 93 - 40	Z11.7-1940
Analysis of Grease	D 128 - 40	Z11.16-1940
Test for Knock Characteristics of Motor Fuels	D 357 - 40	Z11.37-1940
Test for Water in Petroleum Products and Other Bituminous Materials	D 95 - 40	Z11.9-1940
Test for Water and Sediment in Petroleum Products by Means of Centrifuge	D 96 - 40	Z11.8-1940
Test for Precipitation Number of Lubricating Oils	D 91 - 40	Z11.30-1940
APPROVAL AS AMERICAN STANDARD		
Test for Unsulfonated Residue of Plant Spray Oils	D 483 - 40	Z11.41-1940
Specifications for Stoddard Solvent	D 484 - 40	Z11.42-1940
Title	**ASTM Designation**	**Former ASA Number**
WITHDRAWAL OF APPROVAL AS AMERICAN TENTATIVE STANDARD		
Test for Sulfur in Petroleum Oils by Lamp Method	D 90 - 34 T	Z11.38-1935
Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter	D 155 - 39 T	Z11.34-1939
Test for Neutralization Number of Petroleum Products and Lubricants	D 188 - 27 T	Z11.12-1928

The computed and experimental values are in good agreement, consequently the subcommittee is recommending the publication as information of a Proposed Method for Converting Kinematic Viscosity to Saybolt Furol Viscosity at 122 F. as shown in Appendix I. The Saybolt Furol viscosities in seconds for the kinematic range of 48 to 400 centistokes are based upon the Herschel equations. Above 400 centistokes the relationship between Saybolt Furol viscosity and kinematic viscosity is approximately linear, and the conversion constant of 0.47 is recommended.

TABLE II.—SAYBOLT FUROL AND KINEMATIC VISCOSITIES AT 122 F.

Sample	Viscosity at 122 F.	Laboratory								Average	Average Deviation	Calculated Values[a]
		No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8			
A	Saybolt Furol..........	311.2	310.8	308.0	313.3	309.0	311.3	310.7	...	310.6	1.2	309.5
	Kinematic.............	662.0	660.9	657.4	...	...	...	...	654.7	658.7	2.7	...
B	Saybolt Furol..........	194.8	194.8	194.0	196.9	197.0	195.3	194.9	...	195.4	0.9	194.7
	Kinematic.............	414.5	415.7	413.5	...	...	...	...	411.7	413.8	1.2	...
C	Saybolt Furol..........	106.0	106.6	104.0	107.6	107.0	106.2	106.2	...	106.2	0.7	106.2
	Kinematic.............	225.0	225.2	224.6	...	...	...	...	223.5	224.6	0.5	...
D	Saybolt Furol..........	68.8	69.2	68.0	69.6	69.4	69.1	68.8	...	69.0	0.4	69.0
	Kinematic.............	145.0	145.0	145.0	...	...	...	...	143.6	144.6	0.5	...
E	Saybolt Furol..........	45.4	45.4	45.3	46.0	45.5	45.4	45.0	...	45.4	0.2	45.6
	Kinematic.............	93.8	93.9	93.3	...	...	...	...	93.4	93.6	0.2	...
F	Saybolt Furol..........	32.1	32.2	32.2	32.9	32.3	32.4	32.0	...	32.3	0.2	32.4
	Kinematic.............	64.5	64.6	64.1	...	...	...	...	64.1	64.3	0.2	...
G	Saybolt Furol..........	25.2	25.0	26.2	25.5	25.1	25.2	24.9	...	25.3	0.3	25.0
	Kinematic.............	47.7	47.7	47.6	...	...	...	...	47.4	47.6	0.1	...

[a] The calculations were made in accordance with the following equations:
Below 35 sec. Saybolt Furol:

$$\text{Kinematic viscosity in centistokes} = 2.1\,t - \frac{122}{t}$$

Above 35 sec. Saybolt Furol:

$$\text{Kinematic viscosity in centistokes} = 2.13\,t - \frac{160}{t}.$$

where: t = Saybolt Furol viscosity in seconds.

Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) has been studying for a number of years the variables affecting the results obtained in the determination of neutralization number. This study has shown that temperature, time, degree of agitation, strength and type of solvent, and strength of alkali affect the level and reproducibility of results. Cooperative tests to compare the results obtained by closer control of these variables are shown in Table III.

The subcommittee recommends that the method of test for neutralization number published as information last year,[1] revised to provide for one reheating only, be published as a tentative standard[2] to replace the present Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (D 188 - 27 T). This method was the most reproducible and gave results of the same order as the present tentative method, making possible comparison of previous data.

The subcommittee has prepared a potentiometric method and a so-called "Rapid Method" for determining neutralization number which are published as information only and appear as Appendices II and III, respectively. The potentiometric method permits the accurate determination of neutralization numbers on severely oxidized oils, but since the equipment is not generally available and needs further study, it is

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 309.

[2] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 302.

TABLE III.—NEUTRALIZATION NUMBER BY VARIOUS METHODS.

Laboratory	Rapid Method[a]	Method D 188 — 27 T		Method Published as Information in 1940		Potentiometric Method[b]
		Swirling	Shaking	As Published[1]	With One Reheating[2]	
SAMPLE 4001						
No. 1	0.56	1.09	1.42	1.10	1.00	1.70
No. 2	0.64	...	0.89	1.11	1.04	0.86
No. 3	0.63	0.59	0.99	1.18	1.02	1.09
No. 4	...	...	0.72	0.88	...	0.95
No. 5	...	1.05	...	...	...	...
No. 6	0.47	...	...	1.19	...	0.97
No. 7	...	...	...	...	0.99	...
Average	0.56	0.91	1.00	1.09	1.01	1.11
Average Deviation	0.06	0.21	0.20	0.09	0.02	0.20
SAMPLE 4002						
No. 1	0.23	0.33	0.56	0.33	0.33	0.32
No. 2	0.27	...	0.30	0.35	0.32	0.38
No. 3	0.34	0.27	0.32	0.32	0.30	0.39
No. 4	...	...	0.36	0.41	...	0.34
No. 5	...	0.41	...	...	...	...
No. 6	...	...	...	0.34	0.33	...
No. 7	0.27	...	...	0.37	0.29	0.31
Average	0.28	0.34	0.38	0.35	0.31	0.35
Average Deviation	0.03	0.05	0.08	0.02	0.02	0.03
SAMPLE 4003						
No. 1	0.05	0.05	0.08	0.08	0.08	0.08
No. 2	0.07	...	0.06	0.07	0.07	...
No. 3	0.07	0.03	0.04	0.05	0.05	0.08
No. 4	...	...	0.08	0.07	...	0.11
No. 5	...	0.07	...	...	...	0.20
No. 6	...	...	...	0.04	0.04	...
No. 7	0.09	...	...	0.07	...	0.03
Average	0.07	0.05	0.06	0.06	0.06	0.10
Average Deviation	0.01	0.013	0.02	0.013	0.015	0.044
SAMPLE D 9 - 40 - 1						
No. 1	...	0.44	0.47	0.45	0.42	0.47
No. 2	0.44	...	0.45	0.46	0.45	0.30
No. 3	...	0.40	0.46	0.40	0.40	0.37
No. 4	...	...	0.47	0.50	...	...
Average	0.44	0.42	0.46	0.45	0.42	0.38
Average Deviation	...	0.02	0.008	0.028	0.017	0.06
SAMPLE D 9 - 40 - 2						
No. 1	...	0.88	0.91	0.90	0.85	1.05
No. 2	0.92	...	0.93	0.98	0.96	0.88
No. 3	...	0.91	0.94	0.86	0.97	...
No. 4	...	...	1.02	1.14	...	...
Average	0.92	0.90	0.95	0.97	0.96	0.96
Average Deviation	...	0.02	0.04	0.09	0.07	0.08
SAMPLE D 9 - 40 - 3						
No. 1	...	1.66	1.70	1.66	1.56	1.90
No. 2	1.71	...	1.58	1.73	1.61	1.57
No. 3	...	1.53	1.53	1.53	1.50	1.46
No. 4	...	...	1.29	1.29	...	1.64
Average	1.71	1.60	1.52	1.55	1.56	1.64
Average Deviation	...	0.06	0.12	0.14	0.04	0.13

[a] See Appendix III to this report.
[b] See Appendix II to this report.

not recommended as a tentative standard. The "Rapid Method" is a modification of the method recommended as a tentative standard which shortens the time factor. There are indications that the results of this method may be more significant when studying used crankcase oils, and it is the hope of the subcommittee that by making this method generally available service data may be accumulated.

The subcommittee has also made a comparison of the results obtained by the present Tentative Method of Test for Saponification Number (D 94 - 39 T) and a method using methylethylketone. Since the results of the latter method check results obtained by the present tentative method as shown in Table IV, and since only 10 min. are required to obtain the results, the subcommittee recommends the methylethylketone test as a replacement of the present tentative standard.

TABLE IV.—SAPONIFICATION NUMBER BY PRESENT AND PROPOSED METHODS.

Laboratory	Sample D 4004		Sample D 4005		Sample D 4006	
	Proposed Revision	Method D 94-39 T	Proposed Revision	Method D 94-39 T	Proposed Revision	Method D 94-39 T
No. 1[a]	26.1	26.5	13.2	12.3	...	...
No. 2	26.0	26.1	13.0	13.8	0.43	0.33
No. 3[a]	26.7	26.7	12.7	11.3	0.51	0.51
No. 4	25.9	...	11.9	...	...	...
No. 5	25.4	26.8	14.4	12.6	0.45	0.97
No. 6	26.4	26.9	13.3	12.1	0.70	1.50
No. 7	25.9	26.2	14.4	14.1	0.43	...
Average	26.1	26.5	13.3	12.7	0.50	0.83
Deviation from Average	0.30	0.27	0.66	0.83	0.08	0.41

[a] Refluxing for 30 min.

Subcommittee XXIII on Carbon Residue (L. C. Beard, Jr., chairman) has correlated the results obtained by two methods for determining carbon residue (Conradson Carbon Residue (D 189 - 29), and Ramsbottom Carbon Residue (D 524 - 40 T)) on the basis of available data collected over the past two years

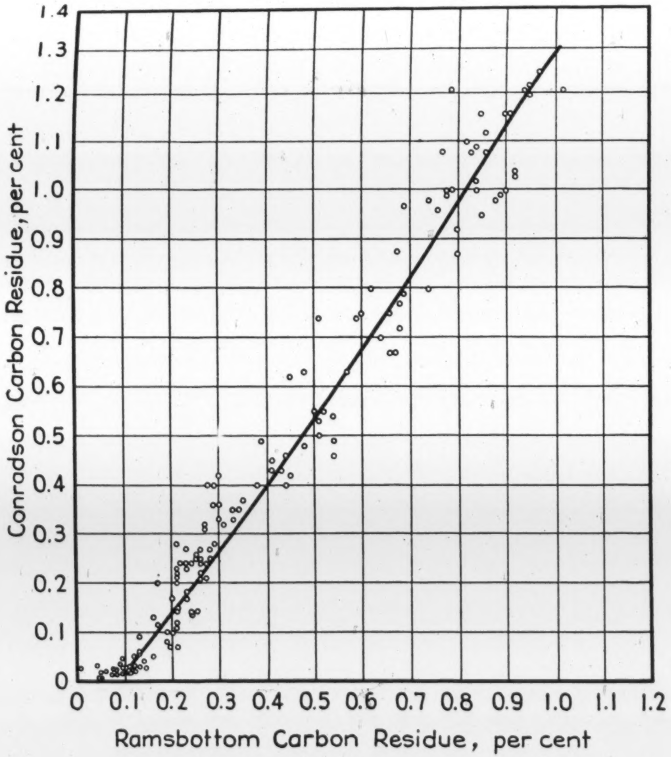

FIG. 1.—Relation Between Ramsbottom Carbon Residue (0.1 to 1.0 per cent) and Conradson Carbon Residue (0.02 to 1.3 per cent).

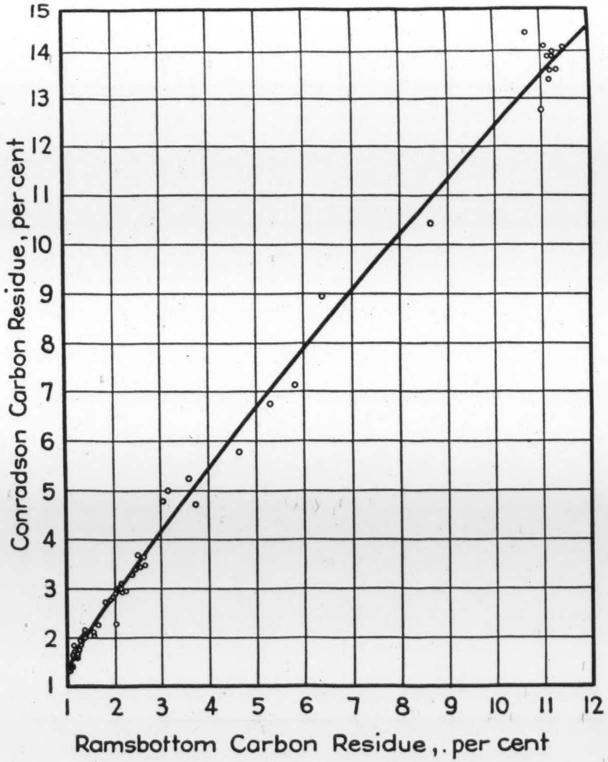

FIG. 2.—Relation Between Ramsbottom Carbon Residue (1.0 to 12 per cent) and Conradson Carbon Residue (1.3 to 14.5 per cent).

amounting to 231 determinations from many different sources on a wide variety of oils. The data are plotted in Figs. 1 and 2, Fig. 1 covering Ramsbottom carbon residues from 0.1 to 1.0 per cent and Fig. 2 those from 1.0 to 12 per cent.

Curves have been drawn through the points on the figures, and equations developed as follows:

$$3.8\,C = R^2 + 4.25\,R - 0.35 \quad (1)$$

$$65\,C = -\,R^2 + 90\,R + 10 \quad (2)$$

where:

C = Conradson carbon residue, and
R = Ramsbottom carbon residue.

TABLE V.—AVERAGE RELATION BETWEEN RAMSBOTTOM AND CONRADSON CARBON RESIDUES, PER CENT.

Ramsbottom	Conradson	Ramsbottom	Conradson	Ramsbottom	Conradson
0.08	0.00	1.20	1.63	4.00	5.44
0.10	0.02	1.30	1.80	4.50	6.08
0.20	0.14	1.40	1.99	5.00	6.70
0.30	0.27	1.50	2.20	6.00	7.90
0.40	0.40	1.60	2.33	7.00	9.10
0.50	0.535	1.70	2.46	8.00	10.25
0.60	0.675	1.80	2.60	9.00	11.35
0.70	0.82	1.90	2.73	10.00	12.43
0.80	0.97	2.00	2.86	11.00	13.50
0.90	1.13	2.50	3.52	12.00	14.55
1.00	1.29	3.00	4.17	13.00	15.60
1.10	1.45	3.50	4.81	14.00	16.50

Equation 1 applies to the range from 0.1 to 1.5 per cent Ramsbottom carbon residue or from 0.02 to 2.2 per cent Conradson carbon residue, and Eq. 2 applies to the range from 1.4 to 12 per cent Ramsbottom carbon residue or from 2.05 to 15 per cent Conradson carbon residue. Equation 1 should not be used for Ramsbottom values below 0.1 per cent as the Conradson values become negative for Ramsbottom values below 0.08 per cent.

Table V shows the numerical relationships between Conradson and Ramsbottom values as taken from the curves in Figs. 1 and 2. It is believed that this correlation is dependable within the combined permissible deviations from the average as specified in the two methods.

Technical Committee A on Gasoline (C. B. Veal, chairman) is recommending the publication as tentative of a Method of Test for Knock Characteristics of Aviation Fuels;[3] the revision of the Tentative Method of Test for Gum Stability of Gasoline (D 525 – 40 T);[4] the revision of the Tentative Method of Test for Tetraethyl Lead in Gasoline (D 526 – 39 T); the reversion to tentative of the Standard Method of Test for Knock Characteristics of Motor Fuels (D 357 – 40); and the publication as information of a Proposed Method of Test for Potential Gum in Aviation Gasoline which appears in Appendix IV.

The revision of Tentative Method D 525 – 40 T consists principally in the reduction of the required sample volume from 100 ml. to 50 ml. and in the addition of a method for correcting to specified temperature the induction periods obtained at slightly different or more convenient operating temperatures. The variation of induction period with temperature may be expressed by the following equation:[5]

$$\log \text{I.P.} = A + \frac{B}{T}$$

where A and B are values characteristic of different gasolines, and T is the absolute temperature.

From the examination of a large number of gasolines, values of B have been found to vary between 9000 and 11,700, averaging about 9900. The average value for six gasolines as reported in 1939 was 10,300. However, for the temperature range permitted in the proposed method (208 to 216 F.)

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[3] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 292.

[4] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 269.

[5] J. W. Ramsay, "Gum Stability of Gasoline," *Industrial and Engineering Chemistry*, Vol. 24, pp. 539-542 (1932); E. W. Aldrich and N. P. Robie, "The Gum Stability of Gasoline," *Journal*, Soc. Automotive Engrs., Vol. 30, pp. 198-207 (1932); Report of Section on Gum, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 400 (1939).

the correction of observed induction periods to induction periods at 212 F. may be made approximately by use of a factor, $1 + 0.056\,\Delta t$, where Δt is the difference in degrees Fahrenheit between the test temperature and 212 F. as outlined in the method. The maximum error introduced by the use of this simplified temperature correction factor on induction periods of 4 hr. may amount to about 4 per cent for gasolines having extreme B values and for tests conducted at the maximum permissible difference from the standard temperature. Ordinarily it will be much less.

The proposed Method of Test for Potential Gum in Aviation Gasoline is essentially a combination of the proposed revision of the Tentative Method of Test for Gum Stability of Gasoline (D 525 - 40 T) and the Standard Method of Test for Gum Content of Gasoline (D 381 - 36) with provision for correction in case the gasoline contains tetraethyl lead.

Technical Committee F on Diesel Fuel Oils (C. G. A. Rosen, chairman) recommends that the Proposed Method of Test for Ignition Quality of Diesel Fuels, published as information last year, be published as a tentative standard,[6] and also that the Diesel-Fuel-Oil Classification published as information in the 1940 report be revised as shown in Appendix V.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following four methods and one specification be accepted for publication as tentative:[7]

Test for Aniline Point of Petroleum Products, essentially as published as information in the 1940 report of the committee.

Test for Carbonizable Substances in Paraffin Wax, similar to the present Tentative Method of Test for Carbonizable Substances in White Mineral Oil (D 565 - 40 T).

Test for Ignition Quality of Diesel Fuels, essentially as published as information in the 1940 report of the committee.

Test for Knock Characteristics of Aviation Fuels, developed by the Cooperative Fuel Research Committee, and

Specifications for Aviation Gasolines, revised form of the specifications published as information in the 1939 report of the committee.

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends revisions of seven tentative standards as follows:

Tentative Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 40 T):[8]

The addition of an appendix containing two equations and a table showing the relationship between values obtained by this method and those obtained by the Conradson carbon residue test (D 189 - 39) is recommended by Subcommittee XXIII on Carbon Residue (L. C. Beard, Jr., chairman) with a reference to the appendix in the present note under Section 1. The equations and table are included in the report of Subcommittee XXIII.

Tentative Method of Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (D 565 - 40 T):[8]

The following clarification of the scope is recommended by Subcommittee I on Pharmaceutical Tests (V. F. Beyer, chairman):

Section 1.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 1. This method of test is applicable to white mineral oil (liquid petrolatum) to ascertain whether it conforms to the standard of quality [prescribed by the U. S. Pharmacopoeia] ***required for pharmaceutical use.***

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[6] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 273.

[7] These methods and specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 257, 263, 273, 292, 251, respectively.

[8] 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 198, 201.

Tentative Method of Test for Gum Stability of Gasoline (D 525 – 40 T):[9]

A revision of this method, as mentioned earlier in this report, is recommended by Technical Committee A on Gasoline (C. B. Veal, chairman). The revised method is appended hereto.[3]

Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (D 188 – 27 T):[10]

A revision of this method, as mentioned earlier in the report, is recommended by Subcommittee XIII (H. P. Ferguson, chairman). The revised method is appended hereto.[2]

Tentative Method of Test for Saponification Number (D 94 – 39 T):[11]

A revision of this method, as mentioned earlier in the report, is recommended by Subcommittee XIII (H. P. Ferguson, chairman). The revised method is appended hereto.[12]

Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (D 90 – 34 T):[13]

A revision of this method is recommended by Subcommittee VII (H. M. Hancock, chairman) to increase its accuracy. The principal features of the revision are a provision for purifying the air used for combustion from acid or acid-forming impurities, and the use of a sintered glass plate in the place of glass rods or beads in the absorber to increase its efficiency. The revised method is appended hereto.[14]

Tentative Method of Test for Tetraethyl Lead in Gasoline (D 526 – 39 T):[15]

The following minor revisions are recommended by Technical Committee A on Gasoline (C. B. Veal, chairman):

Sections 2 and 4.—Delete the capital letters *A*, *B*, *C*, and *D* from Sections 2 (*a*) to (*d*), and from 4 (*a*).

Section 2 (c).—Change "bead" to "line."

Section 2 (g).—Delete "calibrated for gasoline delivery" and footnote 2.

Section 4 (b).—Add the following as Note 4 with reference at the end of the first sentence:

> NOTE 4.—To reduce the evaporation time it is permissible to employ an air jet under the following conditions: Substitute a 500-ml. Erlenmeyer flask for the 400-ml. beaker; evaporate on a hot plate whose surface temperature is maintained between 230 and 260 C., while impinging upon the surface of the liquid a stream of hot (about 75 C.) clean air at a rate of about 10 l. per min. The air stream should be led into the flask by means of a glass tube with an orifice about 5-mm. in diameter, placed about 60 mm. above the surface of the liquid. The air stream should not be used in the nitric acid evaporation.

Section 5 (c).—Insert the words "saturated with lead sulfate" after "cold dilute sulfuric acid" in the next to the last sentence.

Round off the following factors as indicated:

	Change From	To Read
Section 3	0.64108	0.6411
Section 3 and Section 5(*a*)	2.7924	2.792
Section 5(*b*)	45.915	45.92
Section 5(*c*)	48.935	48.94

III. REVISIONS OF STANDARDS

The committee recommends for immediate adoption revisions in three standard methods of test, as set forth below, and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 – 39):[16]

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[9] 1940 Supplement to Book of A.S.T.M Standards, Part III, p. 211.

[10] 1939 Book of A.S.T.M. Standards, Part III, p. 617.

[11] 1939 Book of A.S.T.M. Standards, Part III, p. 620.

[12] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 305.

[13] 1939 Book of A.S.T.M. Standards, Part III, p. 626.

[14] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 308.

[15] 1939 Book of A.S.T.M. Standards, Part III, p. 630.

[16] 1939 Book of A.S.T.M. Standards, Part III, p. 98.

The following additions are recommended by Subcommittee XXIII on Carbon Residue (L. C. Beard, Jr., chairman):

Section 1.—Add a note to read as follows:

> NOTE.—Values obtained are in general not numerically the same as the Ramsbottom carbon residues as determined in accordance with the Tentative Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (A.S.T.M. Designation: D 524). See Appendix.

Appendix.—Add an appendix containing two equations and the accompanying Table V, showing the relationship between the values obtained by the two methods, as included in the report of Subcommittee XXIII.

Standard Method of Test for Distillation of Crude Petroleum (D 285 – 36).[17]

The following revision is recommended by Subcommittee XXI on Crude Petroleum (A. J. Kraemer, chairman) to make the description of the condenser less restrictive without affecting the results:

Section 2 (c).—Change from its present form to read as follows:

> (c) *Condenser.*—A condenser $\frac{9}{16}$ in. (14.29 mm.) in outside diameter made of No. 20 Stubbs gage seamless brass tubing, 22 in. (55.88 cm.) in length. It shall be set so that approximately 15.5 in. (39.4 cm.) of the tube will be in contact with the cooling medium contained in the condenser jacket, subsequently described, with about 2 in. outside the cooling bath at the upper end, and 4.5 in. outside the cooling bath at the lower end. The length of tube projecting at the upper end shall be straight and shall be set at an angle of 75 deg. with the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous, smooth curve. The average gradient shall be 0.259 in. per linear inch of condenser tube (sine of angle of 15 deg.) and no section of the immersed portion of the condenser tube shall have a gradient less than 0.24 in. nor more than 0.28 in. per linear inch of tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 1 to $1\frac{1}{4}$ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle. The capacity of the cooling bath shall be not less than 340 cu. in. (5.55 liters) of cooling medium. The arrangement of the tube in the cooling bath shall be such that its center line shall be not less than $1\frac{1}{4}$ in. below the plane of the top of the bath at its point of entrance and not less than $\frac{3}{4}$ in. above the floor of the bath at its exit. Clearances between the condenser tube and the walls of the bath shall be at least $\frac{1}{2}$ in. except for the sections adjacent to the points of entrance and exit. Multiple installations are permissible, provided they conform to the dimensional requirements and the capacity of the bath is not less than 340 cu. in. per tube.

Standard Method of Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 – 38).[18]

The following revision is recommended by Subcommittee VIII on Distillation (E. W. Dean, chairman) to make the description of the condenser less restrictive without affecting the results:

Section 2 (b).—Make the same change in this paragraph as is recommended above in Section 2 (*c*) of Method D 285.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following three tentative standards be approved for submission to letter ballot of the Society for adoption as standard:

Tentative Method of Test for Distillation of Plant Spray Oils (D 447 – 40 T),[19] on the recommendation of Subcommittee XVII on Plant Spray Oils (J. B. Terry, chairman) revised to make the description of the condenser less restrictive without affecting the results, as follows:

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[17] 1939 Book of A.S.T.M. Standards, Part III, p. 112.

[18] 1939 Book of A.S.T.M. Standards, Part III, p. 123.

[19] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 204.

Section 2 (b).—Make the same change in this paragraph as is recommended above in Section 2 (*c*) of Method D 285.

Tentative Method for Calculating Viscosity Index (D 567 - 40 T),[20] as recommended by Subcommittee V on Viscosity (J. C. Geniesse, chairman), without revision.

Tentative Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 40 T),[21] on the recommendation of Subcommittee XXII on Natural Gasoline (G. G. Oberfell, chairman), revised as follows to improve the wording and introduce a general provision which makes it possible to carry out the test with the initial temperature in the air chamber at 100 F. and eliminate the necessity of correcting for air expansion:

Fig. 1 (c).—Change the dimensions of the bushing in the top of the air chamber from "$\frac{3}{4}$″ x $\frac{1}{2}$″" to "$\frac{3}{4}$″ x $\frac{1}{4}$″" in order to conform to the text of Section 2 (*a*).

Section 14.—In order to insure better handling of samples, change this section to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> 14. *Immediately before proceeding with the Reid vapor-pressure determination the operator shall examine the sample container thoroughly, discarding any sample that had not been securely sealed or whose container shows any evidence of leakage.* [Prior to the Reid vapor pressure determinations, the] *The* sample container shall *then* be immersed in the cooling bath (Section 4), held at a temperature of 32 to 40 F. (0 to 4.4 C.), for sufficient time to allow the liquid to reach the bath temperature. The container shall then be unsealed [for 5 sec.,] *and examined for its liquid content, discarding any sample with a liquid content less than 70 per cent of the container capacity. If the vapor space is less than 20 per cent, the operator shall pour off enough of the sample so that the portion remaining fills the container to not less than 70 nor more than 80 per cent of its capacity. After the proper amount of vapor space has been assured, the container shall then be* resealed, shaken vigorously, and then returned to the bath.

Section 17.—Change Paragraphs (*a*) to (*e*) to read as follows by addition of the italicized words and the omission of those in brackets:

> 17. (*a*) The air chamber shall be rinsed (see Note 4, Section 16 (*c*)) with clean water (temperature optional, *but the determination of the correct "initial air temperature" (Paragraph (d)) will be facilitated if the temperature of the water used for rinsing is within a few degrees of the room temperature adjacent to the air chamber at the time the "initial air temperature" is determined*).
>
> (*b*) The pressure gage shall then be attached to the air chamber.
>
> (*c*) The air thermometer shall next be inserted in the air chamber for three-fourths of its length. The *thermometer* bulb shall not be permitted to rest on the wall of the air chamber. *The thermometer should be supported and held in position midway between the walls of the air chamber by means of a loosely fitting (not airtight) stopper inserted in the opening of the air chamber. Before insertion, the temperature of the thermometer should be within a few degrees of the temperature of the atmosphere adjacent to air chamber at the time of determining the "initial air temperature."*
>
> (*d*) The temperature of the air and water vapor in the air chamber shall be read. *This temperature shall be recorded as the "initial air temperature."* [Since it] *It* is absolutely necessary that [this temperature] *the "initial air temperature"* shall be the actual temperature *plus or minus 0.5 F.* of the air [and] (*saturated with* water vapor) in the air chamber at the [time] *instant* of final assembly (Paragraph (*e*)). *It is imperative that* the air thermometer [shall have been] *remain* in the air chamber (Paragraph (*c*)) [at least 5 minutes] *a sufficient period of time* [before the temperature is read] *in order to obtain accurately the "initial air temperature."* [This temperature shall be recorded as the "initial air temperature."] *If this temperature reading has been constant for 5 min. within 0.5 F., and if this temperature does not differ more than 2 F. from the temperature of the adjacent atmosphere, and if all previous precautions given in this section have been followed, then ordinarily the correct "initial air temperature" has been determined.*
>
> (*e*) The gasoline chamber, immediately (note exception in Section 22) after being filled with

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[20] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 224.

[21] *Ibid.*, p. 214.

the sample (Section 15 [(*b*)] (*d*)), 21 (*b*) or (*c*)), shall be attached to the air chamber with the pressure gage attached (Paragraph (*b*)).

Add the following note after Paragraph (*f*):

Note 6.—Any other procedure for the determination, with an accuracy of 0.5 F., of the temperature of the air (saturated with water vapor) in the air chamber at the instant of final closure of the air chamber prior to vapor pressure determination (Section 18) is permissible.

V. Adoption as Standard of Tentative Revision of Standard

On the recommendation of Subcommittee XXI on Crude Petroleum (A. J. Kraemer, chairman) the tentative revision[22] issued last year of the Standard Method of Test for Distillation of Crude Petroleum (D 285 – 36)[23] is recommended for approval for reference to letter ballot of the Society for adoption as standard.

VI. Reversion of Standard to Tentative

The committee recommends the reversion to tentative status of the Standard Method of Test for Knock Characteristics of Motor Fuels (D 357 – 40)[24] in revised form as recommended by Technical Committee A on Gasoline (C. B. Veal, chairman). The revised method, appended hereto,[25] differs from the present standard in its form and in the addition of requirements for humidity control as in the Proposed Tentative Method of Test for Knock Characteristics of Aviation Fuels.[3] Further revision is expected during the coming year.

TABLE VI.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Test for Aniline Point of Petroleum Products	54	1	7
Test for Carbonizable Substances in Paraffin Wax	43	0	19
Test for Ignition Quality of Diesel Fuels	44	2	16
Test for Knock Characteristics of Aviation Fuels	40	2	16
Specifications for Aviation Gasoline	41	3	18
II. Revisions of Tentative Standards			
Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 40 T)	60	0	2
Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (D 565 - 40 T.	45	0	12
Test for Gum Stability of Gasoline (D 525 - 40 T)	49	1	8
Test for Neutralization Number of Petroleum Products and Lubricants (D 188 - 27 T)	57	3	2
Test for Saponification Number (D 94 - 39 T)	58	1	3
Test for Sulfur in Petroleum Oils by Lamp Method (D 90 - 34 T)	55	1	6
Test for Tetraethyl Lead in Gasoline (D 526 - 39 T)	51	0	11
III. Revisions of Standards			
Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 39), immediate adoption	60	0	2
Test for Distillation of Crude Petroleum (D 285 - 36), immediate adoption	51	0	11
Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 - 38), immediate adoption	51	0	11
IV. Adoption of Tentative Standards as Standard			
Test for Distillation of Plant Spray Oils (D 447 - 40 T), as revised	47	0	15
Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 40 T), as revised	50	0	12
Method for Calculating Viscosity Index (D 567 - 40 T)	55	0	7
V. Adoption as Standard of Tentative Revision of Standard			
Test for Distillation of Crude Petroleum (D 285 - 36)	52	1	9
VI. Reversion of Standard to Tentative			
Test for Knock Characteristics of Motor Fuels (D 357 - 40), as revised	44	1	12

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[22] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 563.

[23] 1939 Book of A.S.T.M. Standards, Part III, p. 112.

[24] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 62.

[25] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 281.

VII. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

The following tentative standards have remained in the tentative status for two years or longer without revision:

Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (D 155 – 39 T).

Test for Consistency of Lubricating Greases and Petrolatum (D 217 – 38 T).

Test for Kinematic Viscosity (D 445 – 39 T).

Test for Sediment in Fuel Oil by Extraction (D 473 – 38 T).

Test for Ash Content of Petroleum Oils (D 482 – 38 T).

Specifications for Fuel Oils (D 396 – 39 T).

It is recommended that these tentative standards be continued in the tentative status because further developments are in progress or in prospect.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 79 voting members, with the results shown in Table VI.

This report has been submitted to letter ballot of the committee which consists of 79 voting members; 62 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

T. A. BOYD,
Chairman.

R. P. ANDERSON,
Secretary.

APPENDIX I

PROPOSED METHOD FOR CONVERSION OF KINEMATIC VISCOSITY TO SAYBOLT FUROL VISCOSITY[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. The conversion table in this method provides a means for converting kinematic viscosity in centistokes at 122 F. to Saybolt Furol viscosity in seconds at 122 F.

Procedure

2. For the kinematic viscosities at 122 F. listed in Table I the equivalent Saybolt Furol viscosity in seconds at 122 F. may be read directly. For kinematic viscosities between 48 and 400 centistokes which are not listed in Table I, the equivalent Saybolt Furol viscosity values may be obtained by linear interpolation. For kinematic viscosities over 400 centistokes, the equivalent Saybolt Furol viscosity may be calculated by use of the multiplication constant given in Table I.

TABLE I.—VALUES FOR CONVERTING KINEMATIC VISCOSITY TO SAYBOLT FUROL VISCOSITY AT 122 F.

Kinematic Viscosity, centistokes	Saybolt Furol Viscosity, sec.	Kinematic Viscosity, centistokes	Saybolt Furol Viscosity, sec.	Kinematic Viscosity, centistokes	Saybolt Furol Viscosity, sec.
48	25.2	100	48.5	200	94.7
50	26.0	105	50.8	210	99.3
52	26.9	110	53.1	220	104.0
54	27.8	115	55.3	230	108.7
56	28.7	120	57.6	240	113.3
58	29.6	125	59.9	250	118.0
60	30.5	130	62.2	260	122.7
62	31.4	135	64.5	270	127.4
64	32.3	140	66.9	280	132.0
66	33.2	145	69.2	290	136.7
68	34.1	150	71.5	300	141.3
70	35.0	155	73.8	310	146.0
72	35.9	160	76.1	320	150.7
74	36.8	165	78.4	330	155.4
76	37.7	170	80.7	340	160.1
78	38.6	175	83.1	350	164.8
80	39.5	180	85.4	360	169.5
82	40.4	185	87.7	370	174.1
84	41.3	190	90.0	380	178.8
86	42.2	195	92.4	390	183.5
88	43.1			400	188.2
90	44.0				
92	44.9			Over 400	Saybolt seconds = centistokes × 0.470
94	45.8				
96	46.7				
98	47.6				

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1941.

APPENDIX II

PROPOSED METHOD OF TEST FOR NEUTRALIZATION NUMBER OF PETROLEUM PRODUCTS BY ELECTROMETRIC TITRATION[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

1. *Scope.*—This method of test is designed to indicate in oxidized petroleum products the presence of constituents having acidic or basic characteristics.

NOTE.—The method is applicable to materials that are soluble, or nearly soluble, in benzene-*iso*propyl alcohol mixtures. Compounds that are only weakly acidic or basic, and whose dissociation constants, K_a or K_b, in water are equal to, or less than $K = 10^{-9}$, are not detectable and do not interfere. Ammonium salts or nitrogen-base salts interfere if their hydrolysis constants are greater than $K = 10^{-9}$. Various groups of acids with different degrees of ionization may be distinguished, provided the dissociation constant of the more strongly acidic group is 10^4 times that of the weaker acidic group.

2. *Neutralization Number:* (*a*) *Neutralization Number.*—Neutralization number is the number of milligrams of potassium hydroxide required to neutralize one gram of sample.

NOTE.—Neutralization number expresses the total amount of the constituents having acid characteristics, and includes the strong acids.

(*b*) *Strong-Acid Neutralization Number.*—Strong-acid neutralization number is the number of milligrams of potassium hydroxide required to neutralize the strong-acid content in one gram of sample.

(*c*) *Base Neutralization Number.*—Base neutralization number is defined as the weight of acid required to neutralize one gram of sample, expressed in equivalent milligrams of potassium hydroxide.

NOTE.—Base neutralization number expresses the total amount of the constituents having basic characteristics, and includes the strong bases.

(*d*) *Strong-Base Neutralization Number.*—Strong-base neutralization number is defined as the weight of acid required to neutralize the strong-base content in one gram of sample, expressed in equivalent milligrams of potassium hydroxide.

3. *Apparatus.*—The apparatus shall consist of the following:

(*a*) *Meter.*—A voltmeter or potentiometer that will operate with an accuracy of plus or minus 0.005 v. and a sensitivity of plus or minus 0.002 v. over the entire range of plus or minus 1.0 v., and will operate on an input of less than 5×10^{-12} amp., when an electrode system having 1000 megohms resistance is connected across the meter terminals. The meter shall be enclosed in a metal shield connected to the ground; a satisfactory terminal shall be provided to connect the shielded connection wire from the glass electrode to the meter without external exposure at any point to electrostatic fields. A continuous reading electronic voltmeter is satisfactory and desirable.

(*b*) *Glass Electrode.*—A pencil-type glass electrode (*C*, Fig. 1) 12.5 to 18.0 cm. (4.9 to 7.1 in.) in length and 0.8 to 1.4 cm. (0.31 to 0.55 in.) in diameter. The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 0.1 to 0.3 cm. (0.039 to 0.117 in.). The end dipping into the solution shall be closed with a hemisphere of Corning 015 glass sealed onto the electrode tube, and the radius of this hemisphere shall be about 0.7 cm. (0.28 in.). The thickness of the glass in the hemisphere shall be great enough so that the resistance of the hemisphere is 200 to 1000 megohms. The electrode shall contain a reproducible, permanently sealed liquid cell for making electrical connection with the inner surface of the hemisphere. The entire electrical connection from the sealed contact cell to the meter terminal shall be surrounded by an electrical shield that will prevent electrostatic interference when the shield is grounded. The shield shall be insulated from the electrical connection by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the electrical connection is greater than 50,000 megohms.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1941.

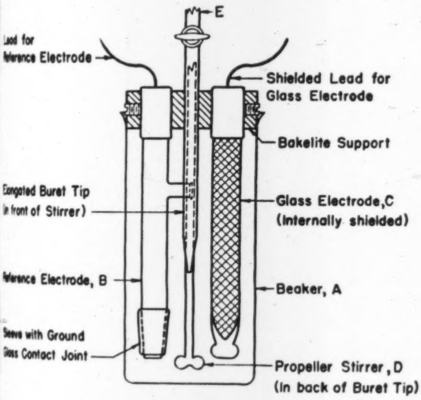
E
Lead for Reference Electrode
Shielded Lead for Glass Electrode
Bakelite Support
Elongated Buret Tip (In front of Stirrer)
Glass Electrode, C (Internally shielded)
Reference Electrode, B
Beaker, A
Sleeve with Ground Glass Contact Joint
Propeller Stirrer, D (In back of Buret Tip)

FIG. 1.—Cell for Potentiometric Titration.

(c) *Reference Electrode.*—A pencil-type calomel electrode (*B*, Fig. 1) 12.5 to 18.0 cm. (4.9 to 7.1 in.) in length and 0.8 to 1.4 cm. (0.31 to 0.55 in.) in diameter. This electrode shall be made of glass and it shall be provided with an external, removable glass sleeve on the sealed end that is dipped into the titration solution. The glass sleeve shall be 0.8 to 2.5 cm. (0.31 to 0.99 in.) in length, shall be slightly tapered, and shall be ground to fit the electrode so that the sealed end of the electrode protrudes 0.2 to 2.0 cm. (0.08 to 0.79 in.) beyond the sleeve. The ground surfaces shall be continuous and free of smooth spots. At a point midway between the extremities of the ground surface, the electrode tube shall be pierced by a hole or holes 0.1 cm. (0.039 in.) in diameter. The electrode shall contain the necessary mercury, calomel, and electrical connection to the mercury, all arranged in a permanent manner. The electrode shall be filled almost to capacity with saturated potassium chloride electrolyte (Section 4 (*f*)) and shall be equipped with a stoppered port through which the electrolyte may be replenished. When suspended in the air and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than 1 drop in 10 min.

(*d*) *Stirrer.*—A variable-speed mechanical stirrer, of any suitable type, equipped with a glass, propeller-type stirring paddle (*D*, Fig. 1). A propeller with blades 1 cm. (0.39 in.) in radius and set at a pitch of 30 to 45 deg. is satisfactory. The stirring apparatus must be electrically correct and grounded so that connecting or disconnecting the power to the motor will not produce an appreciable permanent change in meter reading during the course of the titration.

(*e*) *Burette.*—A burette (*E*, Fig. 1) of 10-ml. capacity, graduated in 0.05-ml. divisions, and calibrated with an accuracy of plus or minus 0.02 ml. The burette shall have a glass stopcock and shall have a tip that extends 10 to 13 cm. (4 to 5 in.) beyond the stopcock.

(*f*) *Beaker.*—A tall-form, lipless glass beaker (*A*, Fig. 1) of 250-ml. capacity, made of chemically resistant glass. The beaker shall be 12 to 13 cm. (4.7 to 5.1 in.) in length, and shall be 5.5 to 6.5 cm. (2.15 to 2.55 in.) in diameter at the open end.

(*g*) *Titration Stand.*—A suitable titration stand to support the electrodes, stirrer, and burette in the position shown in Fig. 1. An arrangement that allows the removal of the beaker without disturbing the electrodes, burette, and stirring paddle is satisfactory.

4. *Reagents:* (*a*) *Standard Potassium Hydroxide (0.1 N in anhydrous isopropyl alcohol).*—Add 6 g. of KOH (A.C.S. analytical reagent grade) to approximately 1 liter of c.p. anhydrous c.p. *iso*propyl alcohol. Boil the mixture gently for 10 min. Add approxi-

mately 2 g.[2] of c.p. barium hydroxide and again boil gently for 5 min. Cool to room temperature, and filter the supernatant liquid through a fine glass filtering funnel. Store the solution in a chemically-resistant bottle, and dispense it in a manner such that it is protected from atmospheric carbon dioxide by means of a guard tube containing soda lime or ascarite and does not come in contact with cork or rubber. Standardize the solution, frequently enough to detect changes of 0.0005 *N*, against standard acid or pure potassium acid phthalate. It is preferable to standardize by potentiometric titration against 0.1600 g. of pure potassium acid phthalate diluted with 125 ml. of CO_2-free water.

(*b*) *Standard Hydrochloric Acid* (*0.1 N in isopropyl alcohol*).—Add 9 ml. of c.p. HCl (sp. gr. 1.19) to 1000 ml. of anhydrous *iso*propyl alcohol. Standardize the solution, frequently enough to detect changes of 0.0005 *N*, against the standard KOH. It is preferable to standardize by potentiometric titration against 8.00 ml. of the standard KOH (Paragraph (*a*)) diluted with 125 ml. of CO_2-free water.

(*c*) *Titration Solvent.*—Add 500 ml. of c.p. benzene and 5 ml. of water to 495 ml. of c.p. anhydrous *iso*propyl alcohol. The titration solvent should be made up in large quantities, and its inherent acidity determined by a blank titration each day prior to its use.

(*d*) *Standard Buffer Solution* (*for standardizing the glass electrode*).—Dissolve 5.105 g. of analytical grade potassium acid phthalate in CO_2-free distilled water, and dilute to exactly 500 ml. This solution must be made up fresh when needed. The pH of this buffer solution is taken as 4.00 at 25 C. and is accepted as being constant at all temperatures between 15 and 35 C. for the purposes of this method; the correct glass electrode potential, cG, of this solution shall be taken as 0.236 at 25 C.

(*e*) *Isopropyl Alcohol.*—C.P. anhydrous *iso*propyl alcohol.

(*f*) *Potassium Chloride Electrolyte.*—Prepare a saturated solution of KCl in water.

5. *Characteristics of Electrode System:*

(*a*) *Preparation for Use and Maintenance.*—The electrode system requires regular attention to obtain constant and reproducible values. Before and after using, wipe the glass electrode thoroughly with a piece of clean cloth, and rinse with distilled water. Also, wipe the calomel electrode with a piece of clean cloth, carefully remove the ground-glass sleeve, and thoroughly clean each ground surface by wiping with the cloth. Flush the ground-glass contact joint (salt bridge) by allowing a few drops of the potassium chloride electrolyte to drain through. Wet the ground surfaces thoroughly with potassium chloride electrolyte, and replace the glass sleeve. With the sleeve firmly in place to eliminate possible leakage, rinse the calomel electrode well, with distilled water.[3] The calomel electrode shall contain, at all times, sufficient saturated potassium chloride electrolyte so that the level of the salt solution inside the calomel electrode is above the level of the liquid in the titration beaker. Although the electrodes are not extremely fragile, they should be handled carefully at all times. When not in used, keep the lower half of each electrode immersed in distilled water. Also, frequently clean the glass electrode by immersion in cold chromic acid solution such as is used for cleaning glass vessels. Once each week drain all the electrolyte from the calomel cell and replace with fresh potassium chloride electrolyte.

(*b*) *Calibration.*—To calibrate the electrodes after preparation, place sufficient standard buffer solution in the beaker to cover the lower half of the electrodes and stir for several minutes while maintaining the temperature of the buffer solution within plus or minus 2.0 C. of the temperature at which the titrations are to be made. Measure the potential, *E* volts, between the electrodes. The cell constant, E_{CG},[4] for the titration cell is calculated from the expression

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[2] Barium hydroxide is insoluble in anhydrous *iso*propyl alcohol, and any excess above that required to react with the carbonate is removed along with the barium carbonate in the subsequent filtration.

[3] Keeping the ground-glass joint free of foreign material is very important in obtaining reproducible potentials in the titration cell, since contamination may introduce uncertain and unnoticeable liquid contact potentials. While this is of secondary importance when titration end points are chosen by inflection points, it may be quite serious when titration end points are taken at fixed cell potentials.

[4] E_{CG} is a constant only in the sense that it combines all voltage effects that should not vary throughout a titration due to a variation in the hydrogen ion activity. For this reason, it must be determined as described at the temperature that is to be used, and redetermined at intervals thereafter.

$$E_{cG} = E - 0.236$$

Then, the correct glass electrode potential, cG, in any subsequent titration made at a temperature within plus or minus 2.0 C. of the calibration temperature, will be the difference between the observed potential, E, at that point and the cell constant, or

$$cG = E - E_{cG}$$

The E_{cG} value of most glass-electrode-calomel-electrode systems will have a value approximately equal to −0.450 v. In the mathematical relations given, E bears the sign indicated by a voltmeter with the glass electrode connected to the negative pole of the meter (the negative pole is the pole to which the zinc terminal of a common dry cell must be connected to give an up-scale reading on the meter).

In the event that the meter is connected to operate as a pH meter, and the pH scale on the meter is being read, vary the so-called "asymmetry potential" adjustment until the meter registers a scale reading, in pH units, equal to 4.00 while the electrodes are immersed in the buffer solution. In this case, the correct glass-electrode potential, cG, in any subsequent titration made within plus or minus 2 C. of the calibration temperature, T, will be directly proportional to the meter reading in pH units and can be calculated as follows:

$$cG = 0.000198\, T \times \text{pH meter reading}$$

where T = calibration temperature in degrees Kelvin.

(*c*) *Performance Test of Electrode System and Meter.*—When dipped into 100 ml. of titration solvent containing 2 to 3 ml. of 0.1 N alcoholic KOH in excess, the emf. between the glass and reference electrodes shall have changed more than 0.30 v. (5 pH units) from the emf. between the same electrodes dipped in an aqueous buffer solution (pH = 7).

6. *Neutralization Number and Strong-Acid Neutralization Number:* (*a*) *Size of Sample.*—The size of sample shall be as follows:

Neutralization Number	Weight of Sample, g.
0.0 to 0.75	20.0 ± 0.05
0.75 to 3.0	5.0 ± 0.02
3.0 to 15.0	1.0 ± 0.005
15.0 to 75.0	0.2 ± 0.001
75.0 to 250	0.1 ± 0.0005

(*b*) *Procedure.*—Weigh or pipette the sample into a 250-ml., tall-form, lipless beaker, and add 125 ml. of titration solvent. Prepare the calomel and glass electrodes as directed in Section 5, and connect the frame of the motor and the meter to a suitable ground to prevent electrostatic interference. Adjust the beaker so that the lower half of each electrode is immersed in the solvent, start the mechanical stirrer, and adjust its speed so that there is vigorous stirring without spattering. Fill the 10-ml. burette with 0.1 N alcoholic KOH, and place the burette in position in the titration assembly so that the tip is immersed approximately 1 in. in the titration solvent. Record the initial reading of the emf. between the electrodes. Add suitable small portions of the 0.1 N alcoholic KOH and, after waiting until an unchanging potential[5] has been established, record the emf. and burette reading. When 0.1 ml. of KOH produces a total change in potential of more than 0.03 v.,[6] add the 0.05-ml. portions. In the region where the addition of 0.1 ml. of KOH changes the potential less than 0.03 v., add a larger portion of KOH sufficient to produce a total change of potential approximately equal to, and not greater than, 0.03 v. Titrate in this manner until the potential changes less than 0.005 v. (corresponding to 0.1 pH units) per 0.1 ml. when the emf. between the electrodes indicates a cG potential greater than 0.65 v. (corresponding to pH scale reading of 11.0). Remove the titrated solution, wash the electrodes well with *iso*-propyl alcohol, and immerse the electrodes in distilled water.

Prepare the calomel and glass electrodes as before and repeat the titration, using 125 ml. of titration solvent but no sample. In this blank titration, add 0.05-ml. portions of 0.1 *N* alcoholic KOH, and wait after each portion until the cell potential does not change noticeably[5] before recording the burette reading and the emf. between the electrodes.

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[5] If the potential changes less than 0.005 v. (corresponding to 0.1 pH unit) per min., it shall be considered constant. This may require from 1 to 3 min. when adding large increments, or when titrating a sample containing less than 0.1 milliequivalent of acidity. When adding 0.05-ml. increments, or when titrating samples containing more than 0.1 milliequivalent of acidity, the normal time of equilibration is generally 1 min. or less.

[6] A change of 0.03 v. corresponds to approximately 0.5 pH units.

Plot a graph with milliliters of KOH as abscissae and millivolts (or pH scale readings) as ordinates. Typical curves are shown in Fig. 2. Mark inflection points that occur in the neighborhood of *cG* potentials of 0.65 and 0.236 v. (corresponding to pH scale readings of 11.0 and 4.0, respectively). Determine the number of milliliters of KOH used to each inflection point.

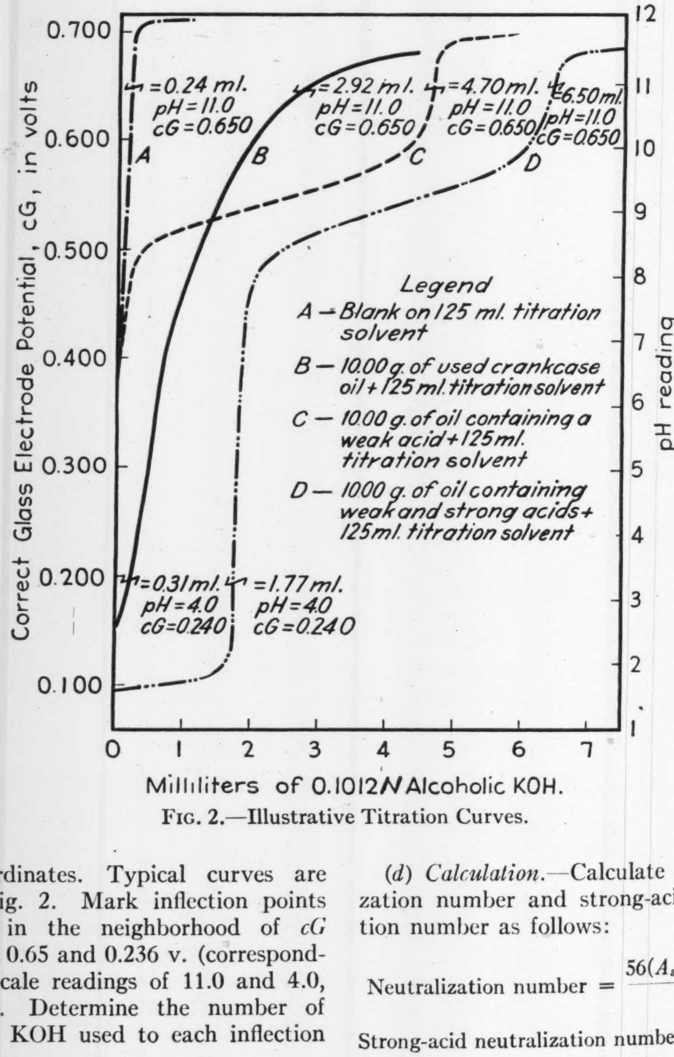

FIG. 2.—Illustrative Titration Curves.

(*c*) *Modified Procedure.*—In those cases in which the titration curve of the sample has no definite inflection point,[7] the titration shall be made as directed in Paragraph (*b*), except that the number of milliliters of KOH used to obtain *cG* equal to 0.24 and 0.65 v.[8] shall be taken as equivalent to the KOH necessary to neutralize the strong acids and the total acids, respectively. The rates of addition of the KOH shall be the same as specified in Paragraph (*b*).

(*d*) *Calculation.*—Calculate the neutralization number and strong-acid neutralization number as follows:

$$\text{Neutralization number} = \frac{56(A_s - A_b) \times N}{W}$$

Strong-acid neutralization number

$$= \frac{56(B_s - B_b) \times N}{W}$$

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[7] Curve *B* in Fig. 2 is an example.

[8] Corresponding to pH scale readings of 4.0 and 11.0, respectively.

where:

A_s = milliliters of KOH required to neutralize the acids up to the end point at which cG is approximately equal to 0.65 v. (corresponding to pH meter reading of 11.0) and

A_b = corresponding value for the blank titration,

N = normality of the KOH,

W = weight of sample in grams,

B_s = milliliters of KOH needed to neutralize the acids up to the first end point at which cG is approximately equal to 0.24 v. (corresponding to pH meter reading of 4.0) and

B_b = corresponding value for the blank titration.

7. *Base Neutralization Number and Strong Base Neutralization Number:* (*a*) *Size of Sample.*—The size of sample shall be the same as specified in Section 6 (*a*).

(*b*) *Procedure.*—Titrate as directed in Section 6 (*b*) and (*c*), except that standard 0.1 *N* HCl shall be used instead of the standard 0.1 *N* KOH. The rates of addition of HCl and the manner of location of end points shall be the same as specified for the titration with KOH in Section 6 (*b*) and (*c*).

(*c*) *Calculation.*—Calculate the base neutralization number and strong-base neutralization number as follows:

$$\text{Base neutralization number} = \frac{56(C_s - C_b) \times N}{W}$$

Strong-base neutralization number

$$= \frac{56(D_s - D_b) \times N}{W}$$

where:

C_s = milliliters of HCl required to neutralize the bases down to the end point at which cG is approximately equal to 0.24 v. (corresponding to pH meter reading of 4.0) and

C_b = corresponding value for the blank titration,

N = normality of the HCl,

W = weight of sample in grams,

D_s = milliliters of HCl required to neutralize the bases down to the end point at which cG is approximately equal to 0.65 v. (corresponding to pH meter reading of 11.0) and

D_b = corresponding value of the blank titration.

8. *Reproducibility of Results.*—(*a*) With proper care, and strict attention to details, duplicate determinations by the same operator should not differ by more than the following amounts:

Type of Titration Curve	Permissible Difference, ml. of 0.1 *N* acid or base
Good inflection point	0.05
No inflection point	0.10

(*b*) Results obtained by different operators may differ by double the amounts given in Paragraph (*a*).

APPENDIX III

PROPOSED METHOD OF TEST FOR NEUTRALIZATION NUMBER OF NEW AND USED CRANKCASE OILS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Phildelphia, Pa.

Scope

1. This method of test is designed to indicate in new and used crankcase oils the presence of organic constituents and metallic compounds having acid or basic characteristics.

NOTE.—Strong acids, if present, shall be determined in accordance with Section 6 (*a*) of the Tentative Methods of Test for Neutralization Number of Petroleum Products and Lubricants (A.S.T.M. Designation: D 188) of the American Society for Testing Materials.[2]

Neutralization Number

2. (*a*) *Neutralization Number.*—Neutralization number is the number of milligrams of potassium hydroxide required to neutralize one gram of sample.

NOTE.—Neutralization number expresses the total amount of strong acid, organic compounds of an acidic nature, and acidic metallic addition agents that will react with potassium hydroxide under the conditions of this test procedure.

(*b*) *Alkali Neutralization Number.*—Alkali neutralization number is defined as the weight of acid required to neutralize one gram of sample, expressed in equivalent milligrams of potassium hydroxide.

NOTE.—Alkali neutralization number expresses the total amount of compounds of an alkaline nature that will react with sulfuric acid within the time limits specified in this procedure.

Apparatus

3. The apparatus shall consist of the following:

(*a*) *Erlenmeyer Flask.*—An Erlenmeyer flask of 250-ml. capacity, made of heat-resistant glass.

(*b*) *Burette.*—A burette of any convenient capacity (5 ml. or greater), graduated in not greater than 0.1-ml. subdivisions.

(*c*) *Balance.*—A platform balance accurate to at least 0.1 g.

(*d*) *Hot Plate.*—A hot plate capable of heating samples from room temperature to boiling within 5 min.

Reagents

4. (*a*) *Aqueous Potassium Hydroxide (1 ml. = 5 mg. of KOH).*—Dissolve 5.1 g. of c.p. potassium hydroxide in 1 liter of freshly boiled and cooled distilled water. Add a very small amount of barium hydroxide, sufficient to precipitate any potassium carbonate present. Standardize this alkali solution against a solution of benzoic acid (prepared from National Bureau of Standards' standard sample 39*e*) in the alcohol described in Paragraph (*c*), using phenolphthalein as the indicator. Titrate without heating. Correct the titration by running a blank on the same amount of alcohol, contain-

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1941.

[2] 1941 Supplement to Book of A.S.T.M. Standards, Part III, 302.

ing no benzoic acid. Adjust the potassium hydroxide solution so that 1 ml. contains 5 mg. of KOH (equivalent to 10.88 mg. of benzoic acid).

NOTE.—Fit the solution bottle with a guard tube of soda lime to prevent access of carbon dioxide. The solution should be restandardized at necessary intervals.

(b) *Diluted Sulfuric Acid (1 ml. = 5 mg. of KOH).*—Adjust a c.p. sulfuric acid solution so that 1 ml. of the acid will be required to neutralize 1 ml. of the standard alkali, using phenolphthalein as indicator, and titrating in a boiling solution.

(c) *Alcohol Solution.*—Dilute one volume of 95 per cent ethanol, to which has been added 10 per cent by volume of methanol (Note), to two volumes with distilled water.

NOTE.—This composition is available under the name of: "U. S. Treasury Dept. Specially Denatured Formula 30 (Regulation No. 3—1938)." Formula 3A plus 5 per cent methanol is an equivalent.

(d) *Phenolphthalein Indicator.*—Dissolve 10 g. of the indicator in 1 liter of either 95 per cent pure ethanol, or 95 per cent ethanol to which 10 per cent by volume of methanol has been added.

Neutralization Number

5. (a) *Procedure.*—Weigh 10 ± 0.1 g. of a representative sample into a 250-ml. Erlenmeyer flask (Note 1). Add 100 ml. of the alcohol solution, agitate by swirling, and heat to boiling within 5 min. Agitate by swirling during the heating process as frequently as may be required to prevent bumping. When boiling begins, remove from hot plate, add 1 ml. of the phenolphthalein indicator, agitate by vigorous swirling for not more than 5 sec., and titrate immediately. Add the KOH solution in increments of 0.1 to 1.0 ml., depending on the rate at which the color fades in the alcohol layer with gentle swirling. After sufficient KOH has been added so that the color does not fade instantly on gentle swirling, add 50 ml. of hot distilled water (175 to 190 F.). Agitate by vigorous shaking (without stopper), tilt the flask at a 45-deg. angle, observe the color of the separated alcohol layer, and, without waiting for a complete separation, proceed with the final titration, adding KOH solution in increments of 0.1 to 0.2 ml. When a definite pink coloration persists, again agitate by vigorous shaking. If the definite pink coloration persists after shaking, the end point has been reached. If too much KOH has been added, the sample may be back-titrated with the diluted H_2SO_4. The entire titration should not consume more than 1 to 1.5 min. for a new or used oil (Note 2).

NOTE 1.—If new oil is being titrated, or if the neutralization number is expected to be quite low, a 20 ± 0.1 g sample may be used.

NOTE 2.—Some samples containing an unusually high percentage of insoluble matter may require a slightly longer time for titration, but in no case should the total titration time exceed 3 min.

(b) *Alcohol Solution Blank.*—Heat 100 ml. of the alcohol solution to boiling, add 1 ml. of the phenolphthalein indicator, and titrate to a definite pink coloration.

(c) *Calculation.*—Calculate the neutralization number of the sample as follows:

$$\text{Neutralization number} = \frac{(A - B) \times 5}{W}$$

where:

A = milliliters of KOH required for titration of sample,
B = milliliters of KOH required for titration of blank, and
W = weight of sample in grams.

Alkali Neutralization Number

6. (*a*) *Procedure.*—If the alcohol layer, on the addition of the phenolphthalein indicator in Section 5 (*a*), develops a pink coloration, titrate rapidly (Note 1) with the diluted H_2SO_4 until the color disappears. Agitate by vigorous shaking (without stopper), tilt flask at a 45-deg. angle, and observe the color in the alcohol layer. If the pink coloration in the alcohol layer does not reappear within 30 sec. (Note 2) after shaking, the end point has been reached.

NOTE 1.—The entire titration should not consume more than 1 min.

NOTE 2.—On standing, the pink coloration may reappear.

(*b*) *Calculation.*—Calculate the alkali neutralization number of the sample as follows:

$$\text{Alkali neutralization number} = \frac{C \times 5}{W}$$

where:

C = milliliters of acid required for titration of the sample, and
W = weight of sample in grams.

Reproducibility of Results

7. With proper care, and strict attention to details, duplicate determinations by the same operator should not differ by more than the amounts given in the following tabulation:

Material Tested	Neutralization Number	Permissible Difference
New oils	not over 0.5	0.05
	over 0.5	10 per cent
Used oils	not over 0.5	0.10
	over 0.5	20 per cent

APPENDIX IV

PROPOSED METHOD OF TEST FOR POTENTIAL GUM IN AVIATION GASOLINE[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

1. *Scope.*—This method describes a procedure for an accelerated oxidation test for aviation gasoline by means of which the quantity of gum is measured after the gasoline has been oxidized for a specified time under prescribed conditions. Provision is made for a correction in the case of aviation gasoline containing tetraethyl lead.

2. *Apparatus:* (*a*) *Bomb.*—The bomb shall be made of a stainless steel (Note 1) with the inside dimensions of the portion that encloses the reacting gasoline-oxygen mixture conforming substantially to those shown in Fig. 1. The interior surface of the bomb and lid shall be given a high polish to facilitate cleaning and to prevent corrosion. Other structural details such as the method of closure, gasket material, and outside dimensions are optional, provided the following limitations are observed:

(*1*) The bomb shall be constructed to withstand a working pressure of 180 psi. at 212 F. (100 C.), with an ultimate strength at least equal to that of a bomb constructed of 18 per cent chromium, 8 per cent nickel alloy steel (Note 1) and having dimensions as shown in Fig. 1.

(*2*) The closure (Note 2) shall be capable of making a seal that will not leak when the bomb is filled with oxygen to 100 psi. at 60 to 80 F. (15.6 to 26.7 C.), and plunged into a bath at 212 F. (100 C.). Any suitable gasket material may be used that will not react with oxygen (Note 3).

(*3*) The weight of the bomb assembled as shown in Fig. 1, but without the oxygen valve and glass liner, shall be not less than 5.0 lb. nor more than 8.0 lb. This weight shall be distributed about the sample container in a manner equivalent to that shown in Fig. 1.

NOTE 1.—A suitable material is an 18 per cent chromium, 8 per cent nickel alloy steel conforming to grade S, type 304 of the Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A.S.T.M. Designation: A 240) of the American Society for Testing Materials;[2] or S.A.E. Nos. 30905 or 30915.[3]

NOTE 2.—It is preferable that the closure ring be constructed from an alloy different from that used for the bomb body, particularly if the bomb metal is that specified in Note 1 and if the mating threads of the two parts are to move with respect to each other when a tightening load is applied.

NOTE 3.—Reactivity with oxygen shall be determined by the following test: The gasket under test shall be placed in the bomb in the absence of gasoline and a gasket of similar composition used in the attachment of the cover. The bomb shall be filled with oxygen to a pressure of 100 psi. and immersed in a bath at about 212 F. (100 C.). If the pressure does not drop more than 2 lb. from the maximum in a 24-hr. period, with the bath temperature differing by not more than 2.0 F. from that when the pressure was at its maximum, the gasket shall be considered nonreactive in the presence of oxygen.

(*b*) *Bomb Accessories.*—The bomb lid shall be provided with a stem and stem filler rod, both constructed of stainless steel and having dimensions substantially as shown in Fig. 1. The inside of the stem and the outside of the filler rod shall be given a high polish to facilitate cleaning. Means shall be provided for connecting to the bomb stem an indicating or recording pressure gage (Paragraph (*c*)) and a tightly closing needle valve (Note 4) through which oxygen may be admitted. The connection to the source of oxygen may be made as indicated in Fig. 1, or it may be made through a tee or cross connection on the top of the bomb stem. A circular metal plate of a diameter sufficient to serve as a lid for closing the bath when the bomb is in place shall be fitted to the stem in any convenient manner but in the position shown in Fig. 1. A glass liner shall be provided, the shape and dimensions of which shall conform to those shown in Fig. 1. The liner shall fit snugly inside the bomb. A suitable holder for the bomb and an adequate wrench for obtaining a tight closure shall be provided.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1941.

[2] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 64.

[3] 1941 S.A.E. Handbook, p. 307.

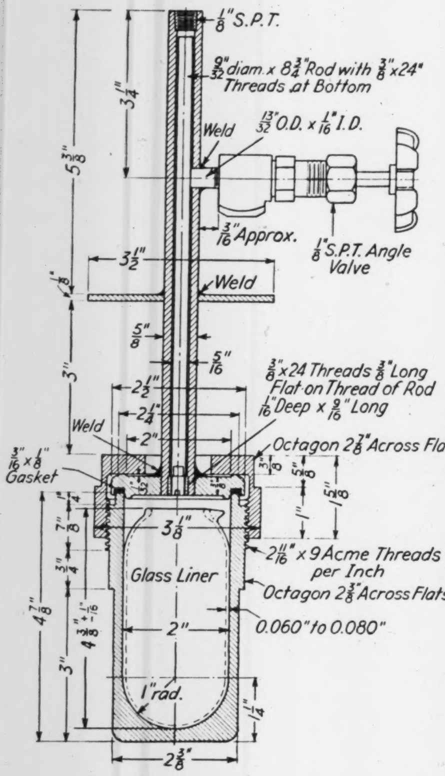
1/8" S.P.T.
9/32" diam. x 8 3/4" Rod with 3/8" x 24" Threads at Bottom
13/32" O.D. x 1/16" I.D.
Weld
3 1/4"
5 3/8"
3/16" Approx.
1/8" S.P.T. Angle Valve
3 1/2"
Weld
1/8"
5/8"
5/16"
3"
3/8" x 24 Threads 3/8" Long
Flat on Thread of Rod 1/16" Deep x 9/16" Long
2 1/2"
2 1/4"
2"
Weld
Octagon 2 7/8" Across Flats
3/16" x 1/8" Gasket
3/8"
5/8"
1 5/8"
1"
1/32"
1/8"
1/4"
7/8"
3 1/8"
2 11/16" x 9 Acme Threads per Inch
Glass Liner
Octagon 2 3/8" Across Flats
3/4"
4 7/8"
4 3/8" ± 1/16"
2"
0.060" to 0.080"
3"
1" rad.
1 1/4"
2 3/8"

FIG. 1.—Bomb for Accelerated Oxidation.

NOTE 4.—It is suggested, for the sake of convenience, that a tire valve and stem be attached to the needle valve and that the oxygen be introduced through a suitable high pressure air hose and fitting. A shortened form of Schrader valve stem No. 724 is satisfactory. The valve stem should not replace the needle valve as the latter serves as a safety closure during the test.

(*c*) *Pressure Gage.*—A 200-psi. maximum pressure gage of either the indicating or recording type shall be used having a precision of not more than 50 psi. per inch of travel of the pointers along the arc of the scale throughout the range of 100 to 180 psi. and having divisions on the chart or scale not greater than 5 psi. The gage may be connected to the bomb stem directly or by a flexible metal tubing, but the total volume of the stem with filler in place and all connections shall be not more than 15 ml.

(*d*) *Oxidation Bath.*—The oxidation bath shall be a water bath having a capacity of not less than 4.5 gal. per bomb, and of such dimensions that the maintained depth of liquid is not less than 11.5 in. The bath shall be equipped with a condenser and a heating unit for keeping the water boiling continuously. It also shall have a thermometer well such that 206 F. (96.7 C.) point of the thermometer (Paragraph (*e*)) shall be of the same level as the upper surface of the bath cover. A round opening of such a diameter as to accommodate the bomb and to fit the plate fastened to the bomb stem shall be provided. The top of the bomb lid shall be submerged at least 2 in. below the surface of the bath liquid.

(*e*) *Thermometer.*—An A.S.T.M. Saybolt Viscosity Thermometer, graduated in either Fahrenheit or Centigrade degrees as specified, having a range of 204 to 218 F. or 95 to 103 C. and conforming to the requirements for this thermometer as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.[4]

(*f*) *Evaporation Bath.*—The evaporation bath shall consist of a closed vessel provided with a reflux condenser, a well into which the beaker fits, a coil for preheating the air, and a conical adapter to deliver the air into the center of the beaker. A suitable form of apparatus is shown in Fig. 2. A multiple bath having similar provisions is also satisfactory. The bath shall be filled to within 1 in. of the top with a stable liquid having a boiling point within a temperature range from 320 to 330 F. (160 to 165.8 C.). Ethylene glycol containing approximately 3 per cent water is suitable for the purpose. The bath shall be insulated with asbestos. An electric hot plate capable of keeping the liquid boiling actively is the preferred heating means. In the absence of a satisfactory hot plate, the bath may be heated by a gas burner, in which case the apparatus shall be placed in a hood with a good draft.

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[4] 1939 Book of A.S.T.M. Standards, Parts II and III.

(*g*) *Catalyst.*—The catalyst shall be clean sand blasted steel (Note 5) in the form of tubing, or strips twisted into spirals to avoid flat contact.

NOTE 5.—A suitable material is carbon steel conforming to grade 3 or 5 of the Standard Specifications for Commercial Quality Hot-Rolled Bar Steels (A.S.T.M. Designation: A 107) of the American Society for Testing Materials;[5] or S.A.E. No. 1020.[6]

(*h*) *Beakers.*—The beakers shall be of the Berzelius type, made of heat-resistant glass, without lip, and of 100-ml. nominal capacity.

(*i*) *Gooch Crucible.*—A sintered-glass Gooch crucible, fine porosity, of 25-ml. nominal capacity shall be used.

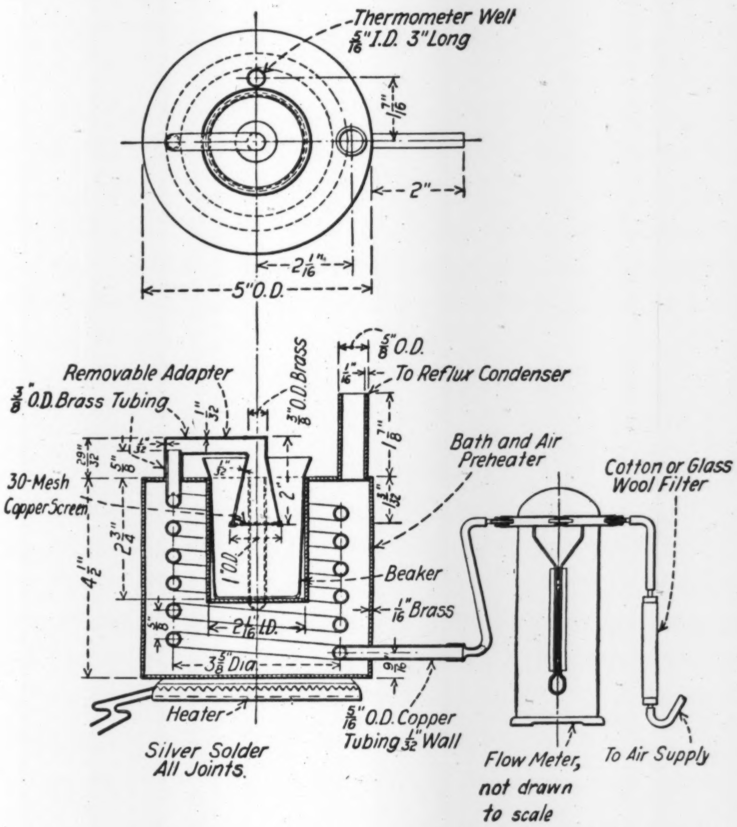
Thermometer Well $\frac{5}{16}$" I.D. 3" Long
$\frac{7}{16}$"
2"
$2\frac{1}{16}$"
5" O.D.
$\frac{5}{8}$" O.D.
$\frac{1}{16}$"
To Reflux Condenser
Removable Adapter
$\frac{3}{8}$" O.D. Brass Tubing
$\frac{1}{32}$"
$\frac{3}{8}$" O.D. Brass
$\frac{29}{32}$"
$\frac{1}{32}$"
$\frac{5}{8}$"
$\frac{1}{32}$"
30-Mesh Copper Screen
2"
$1\frac{7}{8}$"
$1\frac{3}{32}$"
Bath and Air Preheater
Cotton or Glass Wool Filter
$4\frac{1}{2}$"
$2\frac{3}{4}$"
1" O.D.
Beaker
$\frac{1}{16}$" Brass
$\frac{5}{8}$"
$2\frac{1}{16}$" I.D.
$3\frac{5}{8}$" Dia.
$\frac{9}{16}$"
Heater
$\frac{5}{16}$" O.D. Copper Tubing $\frac{1}{32}$" Wall
Silver Solder All Joints.
Flow Meter, not drawn to scale
To Air Supply

FIG. 2.—Apparatus for Determining Gum Content of Oxidized Gasoline.

(*j*) *Air Supply.*—Low-pressure air shall be passed through a cotton or glass-wool filter and delivered to the preheater inlet of the bath. A flowmeter or other air-measuring device shall be provided as shown in Fig. 2. The flowmeter shall be calibrated when attached to the bath with the bath liquid at room temperature.

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[5] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 9.

[6] 1941 S.A.E. Handbook, p. 304.

3. *Procedure.*—(*a*) Carefully clean the bomb and its accessories before starting the test. Wash the glass liner with a suitable gum solvent (Note 6) and allow to dry. Soak the liner for a minimum of 12 hr. in chromic acid solution, rinse three times with distilled water, and finally dry by heating in an oven. The glass liner should be handled only with clean metal forceps after cleaning. Drain from the bomb any gasoline present and wipe the inside of the bomb and cap, first with a clean cloth moistened with the gum solvent and then with a clean dry cloth. Remove the gum or gasoline in the annular space with gum solvent. Occasionally remove the filler rod from the stem and carefully clean both stem and rod. The annular space shall be thoroughly dry before each test is started.

NOTE 6.—Either acetone or a blend of benzol-ethyl alcohol (1:1) is a satisfactory gum solvent.

(*b*) Bring the bomb and the gasoline to be tested to a temperature of 60 to 80 F. (15.6 to 26.7 C.). Place sufficient clean catalyst (Note 7) of suitable length in the glass liner in such a manner that 17½ sq. in. of catalyst surface will be exposed to and completely immersed in the fuel sample. Then add 100 ± 1 ml. of sample to the glass liner containing the catalyst and place the liner in the bomb. Close the bomb (Note 8), introduce oxygen until a pressure of 125 psi. is obtained, and then, after 10 ± 2 min., adjust the pressure between 100 and 102 psi.

NOTE 7.—The catalyst shall be sandblasted to a uniform gray color, washed in HCl (2 per cent), rinsed with distilled water and then with acetone, and stored in kerosine.

NOTE 8.—After a new gasket has been inserted in the groove, the bomb cap must be centered carefully so that it will press and seat the gasket properly. When using a new gasket, it is desirable to screw the cap down firmly, release, and retighten several times in order to avoid leaks.

(*c*) Place the charged bomb in the vigorously boiling water bath, being careful to avoid shaking, and record the time of immersion as the starting time. Maintain the temperature of the water at 212 ± 0.2 F. (100 ± 0.11 C.). Leave the bomb in the oxidation bath for 5 hr. (Note 9).

NOTE 9.—If, as indicated by a steady drop in pressure, a leak develops after the bomb is placed in the bath, the test shall be discarded.

(*d*) At the completion of the oxidation period, remove the bomb from the bath and cool rapidly with water while the charging valve is still closed. Release the pressure gradually, take the bomb apart, and remove the glass liner.

(*e*) Drain the oxidized sample from the glass liner into a beaker. To remove any insoluble gum, wash the interior of the liner and the catalyst together twice, using a fresh 10-ml. portion of the solvent (Note 6) for each washing. Add these washings to the oxidized sample, shake the mixture, and filter. Reserve the filtrate for the gum determination (Paragraphs (*f*) and (*g*)).

NOTE 10.—Avoid exposing the sample to daylight prior to and during this test. At the end of the test no visible precipitate shall be present in the glass liner.

(*f*) Wash a test beaker, together with a similar tare beaker, free from gum with the solvent (Note 6), immerse for at least 12 hr. in chromic acid cleaning solution, and wash thoroughly, first with tap water and then with distilled water. Subsequent to removal from the cleaning mixture, the beakers shall be handled only by means of clean metal forceps. After washing, thoroughly dry both beakers by heating to about 302 F. (150 C.), and allow them to cool simultaneously for the same period of time, but not less than 1 hr., in a desiccator. If the air in the balance is dried with a desiccant, this same desiccant shall be used in the desiccator; otherwise no drying agent shall be used in the desiccator. Weigh the test beaker, using its tare (Note 10) on the opposite balance pan. After weighing, replace the tare beaker in the desiccator and place the test beaker in the well of the bath as shown in Fig. 2. Adjust the air flow to a rate, at the point of measurement, of 1 liter per sec., plus or minus 15 per cent. Heat the bath so that the temperature of the effluent air, measured by a thermometer placed on the bottom of the beaker at the center, shall be from 305 to 320 F. (151.7 to 160 C.). This is governed largely by the rate of boiling of the bath liquid and the rate of air flow.

NOTE 11.—For routine tests it is permissible to use the same beaker as a tare for more than one test beaker, provided all are heated at the same time, but not for more than six test beakers.

(*g*) Remove the conical adapter and pour exactly one half of the filtrate plus the washings into the beaker; then replace the adapter on the air outlet so that it is vertically above the center of the surface of the liquid. After the liquid has evaporated, which should require from 8 to 14 min., leave the beaker in place for an additional 15-min. period, the air supply being maintained. During this 15-min. period, heat the tare beaker also to about 302 F. (150 C.). Allow both test and tare beakers to cool simultaneously and for the same period of time, but not less than 1 hr., in a desiccator under the same conditions as prescribed in Paragraph (*f*), and weigh the test beaker as before the test. Report the increase in weight of the test beaker multiplied by 2 as the amount of gum in 100 ml. of the sample.

(*h*) If the gasoline contains tetraethyl lead and the value found under Paragraph (*g*) exceeds 6.0 mg. per 100 ml., treat the residue in the beaker as follows: Add 10 ml. of H_2SO_4 (approximately 95 per cent) to the residue and evaporate the solution to strong fumes of sulfuric acid. Oxidize any organic material by cautiously adding HNO_3 drop by drop until a water-white solution is obtained. Wash this solution into a clean 400-ml. beaker, and add 0.2 to 0.3 g. of $(NH_4)_2SO_4$ to remove completely any HNO_3 that may be present. Fume the sample for a few minutes, remove from the hot plate, cool, dilute to 150 ml., and boil for 5 min. on the hot plate. Remove the beaker from the hot plate, allow to cool, and add 50 to 70 ml. of alcohol. After the solution has cooled to room temperature, filter it through a dried and weighed Gooch crucible prepared as described in Section 3. Wash the precipitate first with cold H_2SO_4 (approximately 2 per cent), and then with alcohol, dry at 110 to 120 C., and weigh as lead sulfate. Multiply this weight by 1.70 and deduct the result from the value found under Paragraph (*g*). Report the resulting figure as the gum in 100 ml. of sample, corrected for tetraethyl lead.

APPENDIX V

DIESEL-FUEL-OIL CLASSIFICATION

INTRODUCTION

Manufacturers and users of Diesel engines, as well as petroleum refiners and distributors, have long felt the need for some classification differentiating types of fuel oils suitable for Diesel engines. The classification shown in Table I, covering four grades of oil, is offered by Technical Committee F on Diesel Fuel Oils of Committee D-2 on Petroleum Products and Lubricants in an attempt to supply this need. This classification was first presented in June, 1934, and has been revised periodically. Criticism and comment should be forwarded to Mr. R. P. Anderson, Secretary of Committee D-2 on Petroleum Products and Lubricants, 50 West 50th Street, New York, N. Y.

SIGNIFICANCE OF CLASSIFICATION REQUIREMENTS

While it is not the intention of the committee that this classification should be used as a purchase specification, a consideration of the significance of the classification requirements should prove helpful in preparing such specifications, particularly where there is a need for a universal diesel fuel suitable for mobile and high-speed equipment. Any such specification, which must take into consideration the availability of base stocks and apply to fuel suitable for a

TABLE I.—DIESEL-FUEL-OIL CLASSIFICATION.

Grade of Diesel Fuel Oil	Flash Point, deg. Fahr.	Pour Point, deg. Fahr.	Water and Sediment, per cent by volume	Carbon Residue, per cent by weight	Ash, per cent by weight	Distillation Temperatures, deg. Fahr.		Viscosity at 100 F.			Sulfur, per cent by weight	Corrosion	Alkali and Mineral Acid	Cetane Number
						90 per cent Point	End Point	Kinematic, centistokes		Saybolt Universal, sec.				
	Min.	Max.	Max.	Max.	Max.	Max.	Max.	Min.	Max.	Max.	Max.			Min.
No. 1-D	100 or legal	0	0.05	...	0.01	...	590	...	...	...	0.5	pass	none	50
No. 2-D	140	20[a]	0.05	0.20[b]	0.01	650	700	2.0 (32.6)[a,c]	6.0 (45.5)[c]	...	1.0	pass	none	45
No. 3-D	140	35[a]	0.1	1.0	0.02	...	...	...	12 (65)[c]	...	1.5	...	none	35
No. 4-D	140	35	0.5	3.5	0.05	...	...	...	...	140	2.0	...	none	30
No. 5-D	Oil more viscous than No. 4-D may be specified by referring to No. 5 fuel oil[d] with an agreement between the purchaser and the seller regarding limits for carbon residue and sulfur.													

[a] Lower pour points may be specified whenever required by local temperature conditions to facilitate storage and use. When operations are anticipated at atmospheric temperatures below an average daily minimum of +10 F., lowering the pour point may also require a lowering of the minimum viscosity limit.

[b] On 10 per cent residuum.

[c] Equivalent values, Saybolt Universal seconds.

[d] Tentative Specifications for Fuel Oils (A.S.T.M. Designation: D 396) of the American Society for Testing Materials.[1]

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[1] 1939 Book of A.S.T.M. Standards, Part III, p. 586.

variety of types of engines, must of necessity be a compromise and it is suggested that No. 2-D be used as a basis.

The following resume of the significance of classification requirements is based upon an analysis of information available from cooperative full-scale engine tests, laboratory data, and field experience by the Automotive Diesel Fuels Division of the Cooperative Fuel Research Committee.

Viscosity:

The importance of viscosity should be recognized as involving a consideration of ease of circulation, atomization, penetration, injection-pump plunger leakage, lubrication quality, heat content, volatility and, finally, over-all power and economy. A lower limit of 2.0 centistokes at 100 F. is considered practical for high-speed diesel engines even though viscosities lower than 2.6 centistokes tend to decrease the power output at a given pump setting because of plunger leakage and inherently lower heat value. When power loss is due to plunger leakage, resetting the pump will, in most cases, restore the maximum power available It is further recognized that when operations are anticipated in a given area at atmospheric temperatures below an average daily minimum of 10 F., the necessity of reaching correspondingly lower pour points may require even a lower viscosity than 2.0 centistokes. An upper limit of 6.0 centistokes should prove practicable for high-speed fuels.

Volatility:

The upper limit of the boiling range will be governed by the requirements of the individual engines. For automotive service the wide speed, load, and temperature variations encountered indicate the desirability of complete distillation of the fuel below 700 F. To insure minimum smoke, odor, lubricating-oil contamination, and engine deposits, it is necessary to avoid an undue concentration of heavy ends in the fuel. Therefore, the temperature at which 90 per cent is evaporated should not exceed 650 F. As the minimum flash point will tend to control the permissible amount of the undesirable low-boiling fractions of the fuel, it is unnecessary to define the lower end of the boiling range.

Flash Point:

Smoothness of combustion and fire hazard are in some way related to front-end volatility and can most conveniently be defined by flash point. Available information indicates that satisfactory operation is possible with fuels having flash points lower than 140 F. and that such a flash point should prove to be a conservative minimum for insuring freedom from operational difficulties.

Carbon Residue:

The fuel property which affects combustion-chamber and nozzle deposits is usually controlled by the volatility limitation. However, to eliminate unusual products which might have excessive concentrations of residue within the allowable volatility range, a carbon-residue requirement on 10 per cent residuum is considered necessary. Performance studies indicate that fuels having a residue not in excess of 0.20 per cent on 10 per cent residuum are satisfactory.

Ignition Quality:

The ease with which a diesel fuel autoignites affects not only its general combustion in the engine but also its starting characteristics. Since most commercially available fuels have cetane numbers less than 60, no upper limit of ignition quality is specified. It is generally recognized that high cetane numbers are desirable, but available fuels meeting the other requirements necessitate the use of a minimum cetane number of 45 for fuel No. 2-D. Information available on a majority of the high-speed engine types used, operating under a wide range of conditions, indicates satisfactory performance on fuels at least as low as 47 cetane number, and this should prove a satisfactory minimum value where a universal fuel is required.

Pour Point:

The pour point of a diesel fuel is of importance in cold weather as it affects handling, pumping, and filtering.

As the best fuels with respect to ignition tend to have poor cold-weather characteristics, the pour point should not be specified lower than absolutely necessary. To obtain adequate supplies of fuels having the other necessary properties that have been outlined, it is advisable that any universal fuel for high-speed diesels have a pour point not lower than 0 F. As the majority of commercially available fuels have cloud points approximately 10 F. above their pour points, and as cloud point limits the flow through the filter, a safe operating temperature for a fuel of 0 F. pour point is 10 F. In those sections where the average daily minimum temperature is below 10 F., it will be necessary to use fuels of correspondingly lower pour point. Under such conditions a corresponding adjustment of the minimum viscosity may be found necessary, as already discussed under *Viscosity*.

Ash:

The Conradson carbon residue limit of 0.20 per cent on 10 per cent residuum will in itself limit the ash content of fuel No. 2-D. However, a separate limitation on ash is desirable, if for no other purpose than to impress upon the user the necessity of avoiding dust and dirt contamination.

Water and Sediment:

Reported experience appears to indicate that a value of 0.05 per cent maximum for water and sediment is satisfactory for high-speed engines.

Sulfur:

Although the evidence regarding the absolute maximum limit for sulfur is inconclusive, experience indicates that a maximum value of 1.0 per cent is satisfactory from the user's standpoint for high-speed engines. Such a limit has the value of permitting the use, in those sections of the country where only high-sulfur fuels can be manufactured, of fuels produced in that territory. A low value would exclude the use of such fuels and thereby limit the total available supply. Higher sulfur limits are indicated for low-speed, heavy-duty engines.

Corrosion:

Heretofore, "passing" of the gasoline corrosion test has been specified for fuels Nos. 1-D and 2-D. As a greater protection against corrosion difficulties in fuel lines of various compositions, "passing" of a 3-hr. copper-strip test at 212 F. is specified for these two grades.

METHODS OF TESTING

The properties listed in this classification are determined in accordance with the following methods of test:

(*a*) *Flash Point.*—Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (A.S.T.M. Designation: D 93),[2] except where other methods are prescribed by law.

(*b*) *Pour Point.*—Standard Method of

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[2] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 47.

Test for Cloud and Pour Points (A.S.T.M. Designation: D 97).[3]

(*c*) *Water and Sediment.*—Standard Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (A.S.T.M. Designation: D 96).[4]

(*d*) *Carbon Residue.*—Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (A.S.T.M. Designation: D 189).[5]

(*e*) *Ash.*—Tentative Method of Test for Ash Content of Petroleum Oils (A.S.T.M. Designation: D 482).[6]

(*f*) *Distillation.*—Distillation of grade No. 1-D oil shall be determined in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86),[7] and of grade No. 2-D oil in accordance with the Standard Method of Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (A.S.T.M. Designation: D 158).[8]

(*g*) *Viscosity.*—Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445),[9] and Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).[10]

(*h*) *Sulfur.*—Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (A.S.T.M. Designation: D 129).[11]

(*i*) *Corrosion.*—Standard Method of Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline (A.S.T.M. Designation: D 130),[12] using a temperature of 212 F. instead of 122 F. The fuel does not "pass" the test if the discoloration of a freshly polished copper strip becomes opaque or if pitting is caused on any portion thereof.

(*j*) *Alkali and Mineral Acid.*—Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (A.S.T.M. Designation: D 188).[13]

(*k*) *Cetane Number.*—Tentative Method of Test for Ignition Quality of Diesel Fuels (A.S.T.M. Designation: D 613).[14]

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[3] 1939 Book of A.S.T.M. Standards, Part III, p. 102.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 82.

[5] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 5.

[6] 1939 Book of A.S.T.M. Standards, Part III, p. 595.

[7] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 35.

[8] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 15.

[9] 1939 Book of A.S.T.M. Standards, Part III, p. 647.

[10] *Ibid.*, p. 216.

[11] *Ibid.*, p. 207.

[12] *Ibid.*, p. 206.

[13] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 302.

[14] *Ibid.*, p. 273.

REPORT OF SECTIONAL COMMITTEE
ON
PETROLEUM PRODUCTS AND LUBRICANTS
ASA PROJECT: Z11

Sectional Committee Z-11 on Petroleum Products and Lubricants held one meeting during the year: in Atlantic City, N. J., on June 27, 1940.

In view of the revision made in the ASA procedure eliminating the status of American tentative standard, approval of the following three A.S.T.M. methods as American tentative standard was withdrawn by ASA on September 30, 1940, on the recommendation of the sectional committee:

Tentative Methods of Test for:

- Sulfur in Petroleum Oils by Lamp Method (D 90 - 34 T; ASA Z11.38—1935),
- Color of Lubricating Oil and Petroleum by Means of ASTM Union Colorimeter (D 155 - 39 T; Z11.34—1939), and
- Neutralization Number of Petroleum Products and Lubricants (D 188 - 27 T; Z11.12—1928).

At the same time, the American Standards Association reapproved eight American standards submitted by the sectional committee in which revisions had been made, and likewise approved two A.S.T.M. standards as American standard for the first time assigning the following ASA designations:

Standard Methods of:

- Test for Distillation of Gasoline, Naphtha, Kerosine and Similar Petroleum Products (D 86 - 40; ASA Z11.10—1940),
- Test for Distillation of Natural Gasoline (D 216 - 40; Z11.11—1940),
- Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 40; Z11.7—1940),
- Analysis of Grease (D 128 - 40; Z11.16—1940),
- Test for Knock Characteristics of Motor Fuels (D 357 - 40; Z11.37—1940),
- Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 40; Z11.9—1940),
- Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 40; Z11.8—1940),
- Test for Precipitation Number of Lubricating Oils (D 91 - 40; Z11.30—1940),
- Test for Unsulfonated Residue of Plant Spray Oils (D 483 - 40; Z11.41—1940), and
- Standard Specifications for Stoddard Solvent (D 484 - 40; Z11.42—1940).

At its meeting held during this annual meeting, the following three A.S.T.M. tentative standards were recommended by Committee D-2 on Petroleum Products and Lubricants for submittal to letter ballot of the Society for adoption as standard. The sectional committee decided to recommend to the American Standards Association the approval of these methods as American standard upon their adoption as A.S.T.M. standards:

Tentative Methods of:

- Test for Distillation of Plant Spray Oils (D 447 - 40 T),
- Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 - 40 T), and
- Calculating Viscosity Index (D 567 - 40 T).

They will accordingly be referred to letter ballot in the sectional committee.

Action was also taken by the sectional committee at this annual meeting to recommend the withdrawal as American standard of the Standard Method of Test for Knock Characteristics of Motor Fuels (D 357 - 39; ASA Z11.37-1939) since this method is being revised and reverted to tentative this year by the A.S.T.M. This recommendation also will shortly be balloted upon by the sectional committee.

Since the standards listed below, already having ASA approval, have been revised this year Committee D-2 has recommended that they be presented to the ASA in their revised form for reapproval as American standard:

Revision of Standard Methods of:

Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 39; ASA Z11.25—1939),
Test for Distillation of Crude Petroleum (D 285 - 36; Z11.32—1936), and
Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 - 38; Z11.26—1938).

The sectional committee voted to refer these revisions to the ASA subject to approval by letter ballot. At the same time a ballot will be taken on the submittal of the Standard Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (D 446 - 39) to the ASA for approval as American standard.

The project on the preparation of specifications for gasoline for use in heavy duty commercial vehicles was discontinued inasmuch as the results of the tests conducted in cooperation with a number of bus operating companies indicated that the problem at the moment is a question of design of fuel systems and is not a question of fuel. The American Transit Assn. accordingly proposes to study further this matter of design in cooperation with vehicle manufacturers to determine whether most of the commercial fuels on the market will not meet all requirements.

Respectfully submitted on behalf of the sectional committee,

T. A. BOYD,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS

Committee D-3 on Gaseous Fuels held two joint meetings with its seven subcommittees, one in Atlantic City, N. J., on October 8, 1940, and a second in Washington, D. C., on March 3, 1941. Four of its subcommittees also met in Washington on March 3, 1941.

Much progress has been made by individual subcommittees in the preparation of proposed tentative standards and several should soon be available. This will be apparent from the brief reports given below on activities of these groups for the past year.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—Equipment has been erected at the National Bureau of Standards and a procedure developed for testing laboratory wet gas meters while in continuous motion. Results obtained by this means are fairly consistent and reproducible. However, there is an appreciable difference between these results and those obtained with the fractional cubic foot bottle. While in all cases so far the direction of this difference has been the same, the magnitude varies somewhat with the different meters. The exact cause of this difference has not yet been established. Further tests are in progress in the hope of obtaining its explanation so that recommendations may be made for a method to be followed in the testing of these meters.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. B. Harper, chairman).—Preparation of a standard method for conduct of heating value determination by use of the flow calorimeter has been largely completed by a subgroup formed for the purpose. Revisions to tables are now being made preparatory to placing the proposed method in final form. When completed, it will cover two types of flow calorimeters in common use and will include procedures for both humidity correction and humidity control. Heating value determinations for gases of 300 Btu. per cu. ft. and over are covered. It is proposed to obtain full information and data on actual use of this proposed method by qualified laboratories prior to its final adoption.

Definitions pertaining to calorific value have been prepared by a second subgroup and are now in the hands of the subcommittee members for approval by letter ballot.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—Tests are still in progress at the National Bureau of Standards on the eleven specific gravity instruments submitted by various manufacturers for that purpose. Although a considerable amount of information is now available on each instrument, it is felt desirable to wait until all the tests have been completed before presenting any actual data. From the information at hand, several of the instruments appear to be capable of surprisingly accurate determinations of specific gravity.

The schedule of test gases calls for the use of 15 test gases covering a range of specific gravities from helium 0.15 to butane 2.0 in approximately uniform steps. The mixtures selected will also give information on the possible effect of variations in compressibility and viscosity as these factors may affect gravity determinations. All tests have been completed on typical synthetic gases with specific gravities of 0.15 to 0.80. Work is now in progress on the eighth test gas which is a mixture of hydrogen and carbon dioxide, having a specific gravity of approximately 0.60. This gas has a viscosity which is very much less than the previous sample tested having approximately the same specific gravity.

It is estimated that all test work will be completed by August 1, 1941, and a general composite report should be available soon thereafter.

Subcommittee V on Determination of Impurities of Gaseous Fuels (E. F. Pohlmann, chairman).—This subcommittee is actively engaged in the preparation of methods for the determination of various impurities in gaseous fuels. Its work is being conducted through three sections, each under the guidance of a leader. Principal attention has been devoted thus far to proposed tentative methods for determination of total sulfur. So far, the subcommittee is not in position to make definite recommendations on methods for determination of individual impurities, although various methods are receiving extensive consideration.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman).—This group is sponsoring active work at Pennsylvania State College on two methods for the measurement of moisture in fuel gases: namely, a laboratory method which depends on the absorption of light by the water vapor at a particular wave length in the near infrared, and a field method which depends on the change of color of cobaltous bromide in an organic solvent on the addition of water. This investigation is described by F. C. Todd and A. W. Gauger in a paper on "Studies on the Measurement of Water Vapor in Gases," to be presented at this annual meeting.[1]

Two meetings of the subcommittee were held last year and progress reports made on the experimental work. The infrared spectrometer and associated equipment has been completed and is at present in operation, but very few data have been obtained as yet. Work has progressed on the colorimetric method until the effect of all the variables on the color are believed to be known and the technique of obtaining the water content from the colorimeter readings has been determined. In the course of this work, a very rough colorimeter was constructed to make a preliminary survey of the problem. Then a precision laboratory model was assembled to determine the possible accuracy of the method. With care, the laboratory model reproduces results with a probable deviation of plus or minus 0.2 per cent of full scale. This laboratory model is not sufficiently strong for field work and has certain basic faults which are being corrected in a new model in the course of construction.

Subcommittee VII on Complete Analysis or Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman).—Analyses of a standard gas sample of the carburetted water gas type have been completed by 24 of the 30 cooperating laboratories and the results reported. These included approximately 225 analyses received from various laboratories employing different apparatus and methods. Results clearly indicate the reproducibility to be expected and offer a direct measure of the accuracy attained in computing the known heating value and specific gravity. Assembled data were discussed at a meeting of the subcommittee held in Washington on March 3. A complete report is being prepared for study and possible action.

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[1] See p. 1134.

A standard sample of the natural gas type will be issued to cooperating laboratories for analysis on return of the sample cylinders.

This report has been submitted to letter ballot of the committee which consists of 33 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

R. M. CONNER,
Secretary.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS

Committee D-4 on Road and Paving Materials has held two meetings during the past year: in Atlantic City, N. J., on June 27, 1940, and in Washington, D. C., on March 6, 1941.

Subsequent to the 1940 annual meeting Committee D-4 presented to the Society, through Committee E-10 on Standards, the following recommendations:

New Tentative Standards:

- Specifications for Cut-Back Asphalt (Rapid Curing Type) (D 597 - 40 T).
- Specifications for Cut-Back Asphalt (Medium Curing Type) (D 598 - 40 T).

Revisions of Tentative:

- Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 - 39 T).
- Specifications for Crushed Stone for Water-Bound Base and Surface Courses (D 489 - 38 T).

These recommendations were accepted[1] by Committee E-10 on August 28, 1940, and the new and revised tentative standards appear in the 1940 Supplement to Book of A.S.T.M. Standards.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee C-4 on Specifications for Emulsified Asphalts (J. E. Myers, chairman).—During the past year this subcommittee has been actively engaged in revising the specifications for slow-setting emulsified asphalt for fine aggregate mixes which were prepared in 1940 but not accepted by Committee D-4. It is hoped that these revised specifications will be ordered to letter ballot of Committee D-4 at its June, 1941, meeting for recommendation to the Society later in the year through Committee E-10 on Standards.[2]

Subcommittee D-1 on Calcium Chloride and Sodium Chloride for Use as Road Materials (H. F. Clemmer, chairman).—This subcommittee has recently submitted to Committee D-4 proposed tentative revisions of Standard Methods of Chemical Analysis of Calcium Chloride (D 345 - 34) and has proposed new tentative specifications for sodium chloride. It is expected that these will be ordered to letter ballot of Committee D-4 at its June, 1941, meeting for recommendation to the Society later in the year through Committee E-10 on Standards.[2]

I. NEW TENTATIVE STANDARDS

As the result of an exhaustive investigation, Subcommittee B-17 on Emulsion Tests (J. S. Miller, chairman), has developed the Tentative Methods of Test for Modified Miscibility and Cement Mixing of Emulsified Asphalts, appended hereto,[3] to be added, when adopted, to the Standard Methods of Testing Emulsified Asphalts (D 244 - 40).[4] The committee recommends that these methods of test be accepted for publication as tentative.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-4 reported the following results of the letter ballot vote from a committee membership of 127: D 597 - 40 T and D 598 - 40 T, affirmative 73, negative 2, ballots marked "not voting" 6; D 75 - 39 T, affirmative 68, negative 3, ballots marked "not voting" 10; D 489 - 38 T, affirmative 69, negative 1, ballots marked "not voting" 11.

[2] See Editorial Note, p. 368.

[3] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 347.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 94.

II. TENTATIVE REVISION OF STANDARD

Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (*C 131 – 39*).[5]—This method of test is under the joint jurisdiction of Committee C-9 on Concrete and Concrete Aggregates and Committee D-4. At the request of Subcommittee IX on Specifications and Methods of Tests of Aggregates (H. F. Clemmer, chairman) of Committee C-9, D. O. Woolf and Stanton Walker reviewed the present standard method of test for the purpose of developing recommendations for revisions providing for the testing of a wider range in gradings of aggregates (see Explanatory Note below). As a result the following revision is recommended:

Section 3 (*b*).—Change from its present form: namely,

(*b*) An abrasive charge of 12 spheres weighing 5000 ± 25 g. shall be used with the grading A described in Section 4, and a charge of 11 spheres weighing 4583 ± 25 g. shall be used with the grading B described in Section 4.

to read as follows:

(*b*) The abrasive charge, depending upon the grading of the test sample as described in Section 4, shall be as follows:

Grading	Number of Spheres	Weight of Charge, g.
A..............	12	5000±25
B..............	11	4584±25
C..............	8	3330±20
D..............	6	2500±15

Section 4.—Change from its present form: namely,

4. The test sample shall consist of 5000 g. of clean, dry aggregate and shall conform to either of the following gradings. The grading used shall be that most nearly representing the aggregate furnished for the work.

Sieve Size (Square Openings) Passing	Retained on	Grading *A* Weight, g.	Grading *B* Weight, g.
1½ in.	1 in..........	1250	0
1 in.	¾ in.........	1250	0
¾ in.	½ in.........	1250	2500
½ in.	⅜ in.........	1250	2500

to read as follows:

4. The test sample shall consist of 5000 g. of clean, dry aggregate and shall conform to one of the following gradings. The grading used shall be that most nearly representing the aggregate furnished for the work.

Sieve Size (Square Openings)		Weight, g.			
Passing	Retained on	Grading A	Grading B	Grading C	Grading D
1½ in.	1 in.........	1250	....	....	....
1 in.	¾ in.........	1250	....	....	....
¾ in.	½ in.........	1250	2500	....	....
½ in.	⅜ in.........	1250	2500	....	....
⅜ in.	No. 3.........	....	....	2500	....
No. 3	No. 4.........	....	....	2500	....
No. 4	No. 8.........	....	....	....	5000

EXPLANATORY NOTE.—The need for a wider range of gradings for use in the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39) has been met in part by the revisions presented this year by Committees C-9 and D-4. The new gradings C and D provide, in particular, for tests of the finer sizes used in concrete and in bituminous surface treatments. However, a further expansion in the range of gradings is needed to permit of tests of larger sizes of concrete aggregates and of aggregates such as are required for penetration macadam, water-bound macadam and other types of construction utilizing the coarser materials.

Gradings A and B, specified in the method as first adopted by the Society in 1937, require abrasive charges of 12 and 11 steel or cast iron spheres, approximately 1⅞ in. in diameter, respectively. Research leading to the development of the test method showed these charges and gradings to give substantially the same results for the same materials. Similarly, the new gradings C and D requiring 8 and 6 of the same spheres have been demonstrated to give approximately the same results. The data for these four gradings indicate the abrasive charges required for similar losses in tests of material from the same source to be a logarithmic function of the surface area of the test sample.

Based on the relationship referred to, the committees proposed at their spring meetings to include requirements for a coarser grading consisting of 2500 g. of 2½- to 2-in. material and 2500 g. of 2- to 1½-in. material tested with an abrasive charge of 15 of the steel or cast iron spheres. Subsequent tests showed the losses thus obtained to be, with minor exceptions, considerably lower than those found

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[5] 1939 Book of A.S.T.M. Standards, Part II, p. 288.

> for the four gradings and charges specified in the proposed revised test method. Also tests made with more than 15 spheres as the abrasive charge failed to show proportionate increases in loss. Accordingly, the recommendation of the coarser grading was withdrawn at the June meeting.
>
> In withdrawing that recommendation the committees instructed the subcommittees having the test method in charge to prepare the preceding statement in explanation of the basis for the revisions and to point to the need for further investigation.

Committee D-4 joins Committee C-9 in recommending that this revision be accepted for publication as a tentative revision of the existing standard.

III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The Committee recommends that the following specifications and methods of test be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 - 39 T),[6]

Tentative Methods of Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 - 39 T),[6] and

Tentative Method of Test for Sieve Analysis of Mineral Filler (D 546 - 39 T).[6]

IV. EDITORIAL CHANGES IN STANDARD AND TENTATIVE STANDARD

Standard Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 - 36).[7]—Committee D-4 recommends that the following addition be incorporated in this standard and accepted as an editorial correction. This change eliminates the possibility of conflict in other requirements of the method and is in the interest of simplification and accuracy:

Section 6 (a).—Change the second sentence to read as follows by the addition of the italicized words:

> The distillation shall be conducted at the rate of between 50 and 70 drops per min., ***except that near the end of the distillation the heat input shall not be so rapid as to result in a temperature in excess of 360 C. (680 F.) after the flame has been removed.***

Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T).[8]—Committee D-4 recommends that the following addition be incorporated in this tentative method and accepted as an editorial correction as the change does not in any way alter the intent or meaning of the method of test but is in the interest of accuracy:

Section 4 (a).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of the word in brackets:

> A 50.0 ± 0.5 g. sample of the water-free material to be tested shall be [weighed] *placed* in[to] a tared container conforming to the requirements of Section 2 (*c*) *and weighed to the nearest 0.01 g. If the material has been heated to facilitate transfer, the sample shall be cooled to room temperature before making this initial weighing.*

Change the last sentence to read as follows by the addition of the italicized words and figures:

> At the conclusion of the heating period the sample shall be removed from the oven, cooled *to room temperature*, weighed *to the nearest 0.01 g.* and the loss due to heating calculated.

V. TENTATIVE STANDARDS CONTINUED AS TENTATIVE

Committee D-4 recommends that the following specifications and methods of test be continued as tentative:

Tentative Specifications for:

Asphalt Filler for Brick Pavements (D 241 - 39 T),
Crushed Slag for Bituminous Concrete Base and Surface Courses (D 485 - 38 T),
Crushed Stone for Bituminous Concrete Base and Surface Courses (D 486 - 39 T),
Crushed Slag for Water-Bound Base and Surface Courses (D 488 - 38 T),
Tar (D 490 - 38 T),

Tentative Method of:

Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T).

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[6] 1939 Book of A.S.T.M. Standards, Part II, pp. 1015, 1017, 1021.

[7] *Ibid.*, p. 394.

[8] 1939 Book of A.S.T.M. Standards, Part II, p. 1045.

Proposed revisions of the following standards and tentative standards have been referred to letter ballot of Committee D-4 for recommendation to the Society subsequent to the annual meeting through Committee E-10 on Standards:[2]

Revisions of:

Tentative Specifications for Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 37 T),
Tentative Specifications for Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 – 39 T),
Tentative Specifications for Crushed Stone for Bituminous Macadam and Base Surface Courses, (D 192 – 39 T), and
Tentative Methods of Sampling Bituminous Materials (D 140 – 39 T).

Tentative Revisions of:

Standard Method of Test for Ductility of Bituminous Materials (D 113 – 39), and
Standard Methods of Chemical Analysis of Calcium Chloride (D 345 – 34).

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Modified Miscibility of Emulsified Asphalt[a]	40	13	40
Test for Cement Mixing of Emulsified Asphalt	48	4	41
II. TENTATIVE REVISION OF STANDARD			
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 39).	67	3	23
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Preformed Expansion Joint Filler for Concrete (Nonextruding and Resilient Types) (D 544 – 39 T)	78	1	5
Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 545 – 39 T)	77	1	5
Test for Sieve Analysis of Mineral Filler (D 546 – 39 T).	80	0	8

[a] The vote on the acceptance as tentative of the Proposed Tentative Methods of Test for Modified Miscibility and Cement Mixing of Emulsified Asphalts (D 244 – 41 T), based on a classification of the results when a division is required was as follows: Affirmative: 9 producers, 20 consumers, 8 general interests; negative: 7 producers, 1 consumer, 2 general interests.

The committee also has under consideration a Proposed Volume Correction Table for Tar and Coal-Tar Pitch which will be presented to the Society for publication as tentative, jointly with Committee D-8 on Bituminous Waterproofing and Roofing Materials, subsequent to the annual meeting through Committee E-10 on Standards.[2]

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 129 members, with the results shown in Table I.

This report has been submitted to letter ballot of the committee which consists of 129 members; 93 members returned their ballots, of whom 81 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. E. MYERS,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-4 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:

Slow-Setting Emulsified Asphalt (for Fine Aggregate Mixes) (D 631 – 41 T),
Sodium Chloride (D 632 – 41 T),

New Tentative:

Volume Correction Table for Tar and Coal-Tar Pitch (D 633 – 41 T), submitted jointly with Committee D-8,

Revision of Tentative Specifications for:

Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 37 T),

Crushed Slag for Bituminous Macadam Base and Surface Courses (D 487 – 39 T),

Crushed Stone for Bituminous Macadam Base and Surface Courses (D 192 – 39 T),

Revision of Tentative Methods of:

Sampling Bituminous Materials (D 140 – 39 T),

Tentative Revisions of Standard Methods of:

Test for Ductility of Bituminous Materials (D 113 – 39), and

Methods of Chemical Analysis of Calcium Chloride (D 345 – 34).

These recommendations were accepted by Committee E-10 on August 25, 1941, and the new and revised tentative standards as well as the tentative revisions appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, pp. 341, 340, 350, 332, 334, 337, 343, 425, and 424.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE

Committee D-5 on Coal and Coke held a meeting in Washington, D. C., on March 4, 1941; another meeting will be held at the annual meeting of the Society in June 1941, in Chicago, Ill.

During the year arrangements were completed for the merging of Sectional Committee M20 on Classification of Coals with Committee D-5. All organizations formerly represented on Sectional Committee M20 that desired representation on Committee D-5 were added to the committee. The scope of Committee D-5 has been enlarged to cover specifications for the classification of coals and now reads as follows:

> *Scope.*—The formulation of methods of sampling, analysis, and testing, and specifications for coal and coke; and of specifications for the classification of coals on the basis of chemical and physical characteristics for domestic and industrial use.

A new subcommittee designated as Subcommittee XVIII on Classification of Coals, under the chairmanship of H. J. Rose, consisting of 21 members, has been formed to have jurisdiction over matters pertaining to coal classification. The standards for coal classification formulated by Sectional Committee M20, which are now under the sponsorship of Committee D-5, are the Standard Specifications for Classification of Coals by Rank (D 388 – 38; A.S.A. M 20.1 – 1938), the Standard Specifications for Classification of Coals by Grade (D 389 – 37; A.S.A. M 20.2 – 1937) and the Standard Definitions for Commercial Varieties of Bituminous and Subbituminous Coals (D 493 – 39; A.S.A. M 20.4 – 1939). The scope of the new subcommittee on coal classification is as follows:

> *Scope.*—The development and revision of specifications and definitions for the classification of all coals from lignite to anthracite to be based upon such chemical and physical characteristics as will make the plan most readily adaptable to industrial and commercial use on a national scale.

In 1939 the committee recommended to the Society for publication as tentative the Method of Test for Index of Dustiness of Coal and Coke (D 547 – 39 T).[1] This year this method is being recommended for adoption as standard.

Because of failure to agree with other committees of the Society interested in the tentative definition of the term coke which has remained on the books of the Society in tentative form for many years, the committee is recommending that this Tentative Definition of the Term Coke (D 121 – 26 T) be withdrawn. The committee believes that any further attempts to formulate a definition of coke should be initiated by Committee E-8 on Nomenclature and Definitions.

Subcommittee VIII on Foundry Coke Specifications has been discontinued as it does not seem to be feasible to prepare general specifications covering foundry coke. The present Standard Specifications for Foundry Coke (D 17 – 16) are being withdrawn as they have outlived their usefulness.

The present membership of Committee D-5 is 53, of whom 14 are classified as producers, 16 as consumers, and 23 as general interest members.

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[1] 1939 Book of A.S.T.M. Standards, Part III, p. 580.

I. ADOPTION OF TENTATIVE STANDARD AS STANDARD

The committee recommends that the Tentative Method of Test for Index of Dustiness of Coal and Coke (D 547 – 39 T)[1] be approved for reference to letter ballot of the Society for adoption as standard.

II. WITHDRAWAL OF STANDARD AND TENTATIVE STANDARD

It is recommended that the Tentative Definition of the Term Coke (D 121 – 26 T)[2] and the Standard Specifications for Foundry Coke (D 17 – 16)[3] be withdrawn for the reasons mentioned earlier in this report.

III. EDITORIAL CHANGE IN STANDARD

The committee recommends that the note in Section 9 of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 40)[4] be deleted when the 1942 Book of A.S.T.M. Standards is published. This note reads as follows:

> NOTE.—For methods of greater accuracy see Report of Subcommittee II on Moisture, of the Joint Committee on Analysis of Coal.

Considerable experimental work on methods of greater accuracy for moisture has been done since the publication of the note in this standard. It is not believed desirable to complicate the standard by reference to such refined analytical procedures as they are hardly practical for use in the purchase and sale of coal.

These recommendations have been submitted to letter ballot of the committee which consists of 53 members; 49 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. ADOPTION OF TENTATIVE STANDARD AS STANDARD			
Method of Test for Index of Dustiness of Coal and Coke (D 547 - 39 T)	37	0	12
II. WITHDRAWAL OF STANDARD AND TENTATIVE STANDARD			
Definition of the Term Coke (D 121 - 26 T)	41	1	7
Specifications for Foundry Coke (D 17 - 16)	38	1	10
III. EDITORIAL CHANGE IN STANDARD			
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 40), elimination of note under Section 9	39	0	10

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman).—A meeting of the subcommittee was held on March 3, 1941, in Washington, D. C. Since trouble is sometimes experienced in determining ash of coals unusually high in calcite and pyrite in getting close agreement between the results of tests made in different laboratories, a special section of the subcommittee was organized to study the subject. This section consists of W. D. Langtry, chairman, C. A. Lunn, and O. W. Rees. At its meeting the subcommittee approved a note prepared by the special section for insertion in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 40) giving instructions for determining ash in these types of coals. This note, which reads as follows, will be presented to Committee D-5 for consideration at the next meeting:

> NOTE.—Difficulty may be experienced in securing satisfactory check determinations of ash in the same or different laboratories for

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[2] 1939 Book of A.S.T.M. Standards, Part III, p. 584.

[3] *Ibid.*, p. 59.

[4] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 5.

coals unusually high in calcite and pyrite. This is caused by varying amounts of sulfate sulfur being retained in the ash. When such difficulty is encountered, or when coals of relatively high ash content whose mineral matter composition is unknown are encountered, the ash should be determined by the following modified procedures:

(*a*) Place the porcelain capsules containing the dried coal from the moisture determination in a cold muffle furnace and heat gradually so that the temperature reaches 500 C. in 1 hr., and 750 C. in 2 hr.. Heat to constant weight at 750 C. By this means pyritic sulfur will be oxidized and expelled before the calcite is decomposed. An ample supply of air in the muffle must be assured at all times to insure complete oxidation of the pyritic sulfur and proper circulation through the muffle must be assured to remove the SO_3 formed.

(*b*) The modified procedure described in Paragraph (*a*) should be adequate for determining ash in all troublesome commercial samples. However, samples may be encountered in certain special studies whose ash values are quite high and whose mineral matter contains much greater than normal amounts of calcite and pyrite. In such cases sulfate sulfur should be determined on the ash obtained by the modified cold muffle method and the value properly corrected, or the Parr[a] sulfated ash method as modified by Rees[b] should be used.

Subcommittee I has taken action recommending for adoption as standard the tentative revision[5] of Section 43 of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 40) which revision pertains to tolerances for the volatile matter determination. This revision is considered of minor nature so will not be presented to the Society until the time of the publication of the 1942 Book of A.S.T.M. Standards.

Experimental work is being conducted on volumetric methods for determination of sulfur in coal and coke. Check determinations of coal ash fusibility are in progress on a series of coal ashes covering a wide range in fusibility using both gas-fired and electrically heated furnaces to obtain data as how best to specify the furnace atmospheres. Trouble has been experienced in some instances when using manufactured gas as fuel to maintain atmospheres as reducing as specified in the tentative revision[5] of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 40).

Subcommittee II on Nomenclature and Definitions (H. C. Porter, chairman).—This subcommittee held a meeting in Washington, D. C., on March 3, 1941. At this meeting it was voted to withdraw the present Tentative Definition of the Term Coke (D 121 – 26 T) which has remained in tentative form for so many years because of failure to formulate a definition acceptable to both Committee D-5 and to Committee D-2 on Petroleum Products and Lubricants. This action was taken in compliance with a request from the Executive Committee of the Society. Committee D-5 has requested Committee E-8 on Nomenclature and Definitions to take the initiative in formulating a definition of coke.

The subcommittee has recommended that Committee D-5 record its approval of the alternative use of the terms "calorific value" and "heat of combustion" to express the quantity of heat developed by combustion and the committee has approved this recommendation. This action by Committee D-5 will necessitate some change in the Tentative Definitions of the Terms Gross Calorific Value and Net Calorific Value of Fuels (D 407 – 35 T) which are being continued as tentative.

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[a] S. W. Parr, "Chemical Study of Illinois Coal," *Bulletin No. 3*, p. 35, Illinois Coal Mining Investigations, State Geological Survey, Urbana, Ill. (1916).

[b] O. W. Rees, "Determining Ash in High Carbonate Coals. Study of the Modified Method," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 9, pp. 307–309 (1937).

[5] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 561.

Subcommittee VII on Pulverizing Characteristics of Coal (John Van Brunt, chairman).—This subcommittee held a meeting on June 27, 1940, in Atlantic City, N. J. Some cooperative work has been done in testing coal grindability by the two tentative methods, namely, the Ball-Mill Method (D 408 – 37 T) and the Hardgrove-Machine Method (D 409 – 37 T), and correlating the results to evaluate the conversion data as given in the Appendix to each of these tentative standards. These tests showed satisfactory agreement between the two methods when using the conversion data as published, but it is believed that more such tests are desirable. The subcommittee has under consideration the feasibility of using the so-called "Lea-Nurse" method[6] for determining specific surfaces of finely sized material as this method might be applicable to grindability tests in measuring the reduction of size of the coal particles in the testing procedures, which is now done by screening methods.

The Tentative Methods of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 37 T) and by the Ball-Mill Method (D 408 – 37 T) are to be continued as tentative.

Subcommittee VIII on Foundry Coke Specifications (J. T. MacKenzie, chairman).—A meeting of the subcommittee was held on March 3, 1941, in Washington, D. C. After much study, the subcommittee has decided that it is not feasible to prepare general specifications for foundry coke so the subcommittee recommended to Committee D-5 that it be dismissed and efforts to prepare foundry coke specifications be discontinued.

The present Standard Specifications for Foundry Coke (D 17 – 16) have outlived their usefulness. The larger consumers of foundry coke prepare their own specifications based on the particular use for the coke. The smaller consumer usually purchases coke, which by trial, has been found to be satisfactory. The subcommittee recommended to Committee D-5 that these standard specifications be withdrawn, and this recommendation was approved by the committee.

Subcommittee XI on Coal Friability (R. E. Gilmore, chairman).—The subcommittee has accumulated data showing the need of revision of the Tentative Methods of Drop Shatter Test for Coal (D 440 – 37 T) and Tumbler Test for Coal (D 441 – 37 T). These tests are therefore being continued as tentative.

Subcommittee XIII on Coal Sampling (H. F. Hebley, chairman).—The subcommittee is continuing as tentative the Method of Sampling Coals Classed According to Ash Content (D 492 – 40 T). It is planned to investigate next methods of sampling coal for determination of total moisture as this determination is of considerable importance. The collection and reduction of samples representing wet coal such as encountered at many cleaning plants requires special precautions to prevent losses of moisture and it sometimes becomes quite a problem as to how such coals can best be sampled for moisture content.

Subcommittee XIV on Dustiness of Coal and Coke (O. O. Malleis, chairman).—This subcommittee recommended for adoption as standard without revision the Tentative Method of Test for Index of Dustiness of Coal and Coke (D 547 – 39 T). Since its publication as tentative in 1939 this method has been used by various laboratories with satisfactory results.

Subcommittee XVI on Ignitibility of Coal and Coke (R. A. Sherman, chairman).—Investigations have been conducted at Battelle Memorial Institute on the various factors that affect a method for determination of ignition temperature of coal and coke developed in the Coal Research Laboratory of the Carnegie Institute of Technology. A paper covering this investigation by Ralph A. Sherman, J. M. Pilcher, and H. N. Ostborg on "A Laboratory Test for the Ignitibility of Coal"[7] is to be presented at this annual meeting of the Society. The test method gives reproducible results which seem to measure an inherent characteristic of a fuel. Further work is in progress to determine whether the ignitibility values can be correlated with burning characteristics of various coals.

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[6] F. M. Lea and R. W. Nurse, "Specific Surface of Powders," *Journal*, Soc. Chemical Industry, Vol. 58, pp. 277-283 (1939).

This report has been submitted to letter ballot of the committee which consists of 53 members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

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[7] Published in ASTM BULLETIN, No. 112, October, 1941, p. 23.

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 25, 1940, in Atlantic City, N. J., and on October 18, 1940, in Washington, D. C.

Cooperative relations are being continued between Committee D-6 and Committee D-9 on Electrical Insulating Materials. Committee D-9 is represented on Committee D-6 by R. W. Chadbourne, and Committee D-6 is represented on Committee D-9 by J. M. Finch. A subcommittee has been formed consisting of three members from Committee D-6 and three members from Committee D-9 that is looking into the question of pH (hydrogen ion concentration) of paper. The three representatives of Committee D-6 to this joint subcommittee are Miss Helen Kiely, Mr. M. S. Kantrowitz, and Mr. J. M. Finch.

In addition, cooperative relations are being carried on between Committee D-6 and Committee C-16 on Thermal Insulating Materials, the Committee D-6 representative being C. C. Heritage.

Committee D-6 is also doing cooperative work with Committee E-1 on Methods of Testing having W. R. Willets as a representative on Technical Committee III on Particle Size and Shape, L. S. Reid on Technical Committee X on Conditioning and Weathering, and M. S. Kantrowitz on Technical Committee XIII on Hydrogen Ion Determinations.

Cooperation between the Society and the Technical Association of Pulp and Paper Industry has been carried out during the year through the Joint ASTM-TAPPI Committee on Paper Testing Methods. G. H. Harnden and R. C. Griffin represent Committee D-6 on this Joint Committee.

During the year D. L. Quinn resigned as chairman of Subcommittee IV on Fiberboard and Fiberboard Containers and R. L. Beach was appointed as the new chairman of this subcommittee. Captain J. M. Berry who formerly represented the National Association of Purchasing Agents, is now the representative of the U. S. War Dept. One member, C. E. Curran was lost by death.

The following additions in membership have been approved during the year:

E. D. Beach, General Fibre Box Co.
K. P. Geohegan, Aetna Paper Co.
C. P. Kirchen, Hollingsworth & Vose Co.
W. B. Lincoln, Inland Container Corp.
R. J. Hemphill, General Electric Co.
P. P. Kennedy, E. I. du Pont de Nemours and Co., Inc.
E. A. Georgi, Hercules Powder Co., Inc.
D. C. Scott, Jr., Henry L. Scott Co.
W. L. Foote, B. F. Perkins Co.,
G. L. Sheldon Nat. Assn. Purchasing Agents.

At the present time the committee consists of 62 members, of whom 53 are voting members; 22 are classified as producer, 17 as consumer, and 14 as general interest members.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following four methods[1] of test be approved for reference to letter ballot of the Society for adoption as standard:

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[1] 1939 Book of A.S.T.M. Standards, Part III, pp. 1051, 1053, 1059, 1055.

Tentative Methods of:
Test for Bulking Thickness of Paper (D 527 - 39 T),
Test for Machine Direction of Paper (D 528 - 39 T),
Test for Water-Soluble Acidity or Alkalinity of Paper (D 548 - 39 T), and
Test for Resin in Paper (D 549 - 39 T).

These recommendations have been submitted to letter ballot of the committee which consists of 53 voting members; 42 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Test for Bulking Thickness of Paper (D 527 - 39 T)	30	0	12
Test for Machine Direction of Paper (D 528 - 39 T)	32	0	10
Test for Water-Soluble Acidity or Alkalinity of Paper (D 548 - 39 T)	29	0	13
Test for Resin in Paper (D 549 - 39 T)	32	0	10

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Paper Testing Methods (W. E. Emley, chairman) has held a number of meetings during the year and has continued its critical study of the various methods of test which have been approved by T.A.P.P.I. and is active in developing new methods of test.

The subcommittee has completed the following four new methods of test, appended[2] to this report as information, which will be submitted to the main committee for approval at the June meeting following which they will be presented to the Society through Committee E-10 on Standards for publication as tentative:

Methods of:
Test for Folding Endurance of Paper,
Test for Basis Weight of Paper,
Test for Thickness of Paper and Paper Products, and
Test for Moisture in Paper.

The four tentative methods which are being recommended for adoption as standard, as mentioned earlier in this report, are under the jurisdiction of this subcommittee.

At the present time methods on the following subjects are being given consideration by Subcommittee I:

1. Absorption
2. Alkali Staining
3. Bursting Strength
4. Conditioning
5. Curl
6. Edge Tear
7. Gloss
8. Hydrogen Ion Concentration
9. Pentosans
10. Ply Adhesion
11. Quantitative Determination of Coating of Paper
12. Saturating Properties
13. Stiffness
14. Tearing Strength
15. Tensile Strength
16. Water Vapor Permeability of Containers, etc.
17. Wire and Felt Sides.

In addition to the above, work has been completed on the following proposed methods which have been turned over to the Joint ASTM-TAPPI Committee for action:

Qualitative Examination of Mineral Fillers and Mineral Coating of Paper,
Air Resistance of Paper.

Subcommittee II on Significance of Test Methods (C. C. Heritage, chairman) held two meetings during the year at which further work was accomplished on the preparation of a monograph covering the significance of test and on definition of terms, nomenclature, and properties of paper. It is planned to include in this monograph a detailed discussion concerning the application of various tests used in evaluating different grades of paper. Information to be included will cover 23 types of tests such as bursting test, tearing test, folding endurance test, etc., on ten different types of paper such a printing paper, wrapping paper, tissue paper, bond, ledger, index, envelope paper, etc. The different members of this subcommittee have been assigned the preparation of various parts of this monograph and these parts are now being received for criticism and final editing.

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[2] See Editorial Note p. 377.

Subcommittee IV on Fiberboard and Fiberboard Containers (R. L. Beach, chairman) has held one meeting during the year. This subcommittee has appointed five special subgroups to make studies on specific test methods such as compression tests, bursting tests, moisture content determination tests, drum test, and puncture test. A Proposed Method for Conditioning and a Method for Compression Test of Fiberboard and Fiberboard Containers have been prepared and approved by the subcommittee. These two methods will be presented to Committee D-6 at the June meeting for approval following which they will be submitted to the Society through Committee E-10 on Standards for publication as tentative.[2]

This report has been submitted to letter ballot of the committee which consists of 53 voting members; 42 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

LEWIS S. REID,
Chairman.

G. H. HARNDEN,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-6 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

- Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (D 641 – 41 T),
- Compression Testing of Corrugated and Solid Fiber Boxes (D 642 – 41 T),
- Test for Folding Endurance of Paper (D 643 – 41 T),
- Test for Moisture in Paper (D 644 – 41 T),
- Test for Thickness of Paper and Paper Products (D 645 – 41 T), and
- Test for Basis Weight of Paper and Paper Products (D 646 – 41 T).

These new tentative methods were accepted by Committee E-10 on August 25, 1941, and assigned the A.S.T.M. serial designations indicated above. These tentative standards appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 584, 586, 589, 594, 596, and 599.

REPORT OF COMMITTEE D-7
ON
TIMBER

Committee D-7 on Timber presents herein a report of its activities during the past year. The committee has held one formal meeting in Chicago, Ill., on March 12, 1941.

This committee has had a tentative specification and a tentative revision of a standard under its jurisdiction, both of which have remained in the tentative status for several years, either because action by cooperating societies was awaited, or because of the necessity for the development of further information. This year these have again been considered, and action was taken at the meeting in Chicago to recommend their adoption as standard as indicated below.

I. Adoption of Tentative Standard as Standard

The Tentative Method of Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions (D 453 - 39 T)[1] has been retained in the tentative status because this method has been under consideration by the Preservatives Committee of the American Wood-Preservers' Association. The A.W.P.A. has now approved and adopted this method as standard. Committee D-7 therefore recommends that the Tentative Method D 453 - 39 T be approved for reference to letter ballot of the Society for adoption as standard.

II. Adoption as Standard of Tentative Revision of Standard

The committee recommends that the definition of water-gas tar, which has been published as tentative for a number of years, be approved for reference to letter ballot of the Society for adoption as standard to be added to the Standard Definitions of Terms Relating to Timber Preservatives (D 324 - 33),[2] with an editorial correction changing the word "flue" to "blue." The definition as recommended for inclusion in the standard reads as follows:

> *Water-Gas Tar.*—Tar produced in the manufacture of carbureted water gas by the decomposition of petroleum oil by heat in the presence of blue gas.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 23 members; 23 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Adoption of Tentative Standard as Standard			
Test for Tar Acids in Creosote and Creosote-Coal Tar Solutions (D 453 - 39 T)	19	0	4
II. Adoption as Standard of Tentative Revision of Standard			
Definitions of Terms Relating to Timber Preservatives (D 324 - 33)	19	0	4

This report has been submitted to letter ballot of the committee which consists of 23 members; 23 members returned their ballots, of whom 22 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

Hermann von Schrenk,
Chairman.

J. A. Newlin,
Secretary.

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[1] 1939 Book of A.S.T.M. Standards, Part II, p. 1126.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 595.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Committee D-8 on Bituminous Waterproofing and Roofing Materials has held two meetings during the past year: in Atlantic City, N. J., on June 27, 1940, and in Washington, D. C., on March 7, 1941.

Subsequent to the 1940 annual meeting, Committee D-8 recommended to the Society through Committee E-10 on Standards the withdrawal of the Standard Methods of Analysis of Roofing Felt for Fiber Composition (D 272 - 34). Reasons therefor were published in an appendix to the 1939 annual report of Committee D-8. This recommendation was accepted by Committee E-10 on August 28, 1940.[1]

Subcommittee IV on Membrane Materials (E. H. Berger, chairman) has recently prepared Proposed Specifications for Coal-Tar Pitch for Steep Built-Up Roofs, Dampproofing, and Waterproofing which will probably be referred to letter ballot for recommendation to the Society for publication as tentative later in the year. The proposed specifications are appended to this report as information.

I. Revisions of Tentative Standards

During the past year the committee has been actively engaged in revising a number of its tentative standards in order to make them more definite and have them conform more closely to accepted good practice. As a result of this work the committee recommends that the following six tentative standards be revised as shown and continued as tentative:

Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 37 T):[2]

Section 6.—Change to read as follows by the omission of the words in brackets:

> (*a*) The surface shall be smooth or [finely] veined in appearance. [Deeply ribbed surfaces shall be rejected.]

Table I.—Change the values for weight per roll to read as follows by the addition of the italicized figures and the omission of those in brackets:

	65-lb. Grade Min., lb.	55-lb. Grade Min., lb.
Weight per roll, net average per 108 sq. ft.....	[62.0] *61.0*	[52.0] *51.0*

Section 10 (a).—Add the following sentence at the end of this paragraph:

> If nails and lap cement are furnished separately, cloth covers or other end protection may be omitted from the ends of the rolls.

Section 11 (a).—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> 11. (*a*) Nails:
>
> (*1*) *Dimensions.*—The gage of the wire shall be not greater than Washburn & Moen No. 10 (0.135 in.) [and not] *nor* less than *Washburn & Moen* No. 12 (0.105 in.). The head shall be not less than $\frac{3}{8}$ in. in diameter, [and not] *nor* less than 0.025 in. in thickness. The shank shall be [between $\frac{3}{4}$ and 1 in. in length, with its lower end pointed] *not less than $\frac{3}{4}$ in. in length* and shall be [either] smooth, [or] barbed, *or square*

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee D-8 reported the results of the letter ballot vote as follows: Of a voting membership of 60, 40 members returned their ballots, of whom 38 voted affirmatively, 0 negatively, and 2 members marked their ballot "not voting."

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 1089.

and twisted (*spiral*). [and if the latter then] *If barbed,* the barbing[s] shall not increase the diameter of the lower two thirds more than 0.01 in. *The lower end of the shank shall be pointed. If square and twisted* (*spiral*), *the cross-sectional area shall be equivalent to that specified above.*

(*2*) *Protective Coating.*—[The] Nails shall be [zinc coated with not less than ½ nor more than 1¼ oz. per sq. ft. of nail surface. Hot-galvanized, electrogalvanized, or sherardized coatings will be accepted] *galvanized or sherardized, and shall resist four 1-min. dips in a copper sulfate solution when tested in accordance with the Standard Method of Test for Uniformity of Coating by the Preece Test* (*Copper Sulfate Dip*) *on Zinc-Coated* (*Galvanized*) *Iron or Steel Articles* (*A.S.T.M. Designation: A 239*) *of the American Society for Testing Materials.*

TABLE I.—PHYSICAL REQUIREMENTS OF ASPHALT SHINGLES.

Shape	The form and size of the shingles shall be as agreed upon by the purchaser and the seller.
Area	Sufficient material per "square" shall be furnished to adequately waterproof 100 sq. ft. of roof surface.
Behavior on heating to 176 F. (80 C.) for 2 hr.	Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than ⅛ in. when suspended vertically.

	Grades			
	Heavy Weight		Regular Weight	
	Max., lb.	Min , lb.	Max., lb.	Min., lb.
Weight, net average per 108 sq. ft.	....	102.0	....	86.0
Weight of dry felt per 108 sq. ft.	....	15.7	....	10.8
Weight of saturant per 108 sq. ft.[a]	....	27.5	....	18.9
[Weight of weather side coating (soluble in CS_2) per 108 sq. ft.]	[20.0]	[12.0]	[20.0]	[12.0]
[Weight of reverse side coating (soluble in CS_2) per 108 sq. ft.]	[5.0]	[2.0]	[5.0]	[2.0]
Weight of weather side mineral filled coating per 108 sq. ft.	*35.0*	*16.0*	*35.0*	*16.0*
Weight of mineral matter per 108 sq. ft. passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve	[40.0]	[25.0] *20.0*	[40.0]	[25.0] *20.0*
[Weight of mineral matter per 108 sq. ft. passing No. 100 (149-micron) sieve]	[10.0]	[4.0]	[10.0]	[4.0]
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of bitumen in the coatings, and the mineral matter passing No. 100 (149-micron) sieve per 108 sq. ft.	*50.0 per cent*	*15.0 per cent*	*50.0 per cent*	*15.0 per cent*
Weight of [complete] roofing per 108 sq. ft	....	97.0	....	83.0

[a] The weight of saturant per 108 sq. ft. shall be not less than 1.75 times the weight of the dry felt.

(*3*) *Quantity.*—There shall be furnished per "square" *an average of* not less than [an average of] 252 nails for 36-in. roofing and not less than [an average of] 275 nails for 32-in. roofing.

New Section.—Add a new Section 14 covering Basis of Rejection to read as follows:

14. *Basis of Rejection.*—Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

Tentative Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules (*D 225 - 37 T*):[3]

Section 1.—Add the following as Paragraph (*b*), relettering the present section as Paragraph (*a*):

(*b*) Shingles supplied under these specifications are intended to be used as follows with a "headlap"[a] of not less than 2 in:

Type of Shingle	Exposure to Weather, in.	Grade
Individual (American Method)	4	Regular
	5	Heavy
Square Tab Strip	4	Regular
	5	Heavy
Hexagonal Tab Strip	4⅔	Regular
	5⅔	Heavy

Individual shingles and shingles of special shapes may also be applied by other methods as agreed upon by the purchaser and the seller.

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[a] The shortest vertical distance between the top of cutout or bottom edge of a course of shingles, and the most proximate underlying area of roof deck not covered by preceding courses of shingles.

[3] 1939 Book of A.S.T.M. Standards, Part II, p. 1092.

Table I.—Change to read as prescribed in the accompanying Table I by the addition of the italicized words and figures and the omission of those in brackets.

New Section.—Add a new Section 13 on Basis of Rejection to read the same as the new Section 14 recommended for addition to Specifications D 224 – 37 T, above.

TABLE II.—PHYSICAL REQUIREMENTS OF ASPHALT ROOFING.

Width	The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.
Area	[The average area of the rolls examined shall contain not less than 108 or 216 sq. ft., as specified, which shall be sufficient to cover 100 or 200 sq. ft., respectively, of roof surface.] *The rolls examined shall contain sufficient roofing to cover 100 sq. ft. of roof surface. (108 sq. ft., with 2 in. or no lapping edge, or 111 sq. ft. with 3-in. lapping edge.)*
Pliability at 77 F. (25 C.)	At least 8 strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ½-in. radius.
Behavior on heating to 176 F. (80 C.) for 2 hr.	Not more than 1.5 per cent volatile matter loss. There shall be no flowing, sagging, blistering, or absorption of the asphalt coatings.

	65-lb. Grade		55-lb. Grade	
	Max., lb.	Min., lb.	Max., lb.	Min., lb.
Weight per roll, net average per 108 sq. ft.	....	62.0	....	52.0
Weight of dry felt per 108 sq. ft.	....	13.5	....	10.8
Weight of saturant (soluble in CS_2) per 108 sq. ft.[a]	....	21.6	....	17.5
[Weight of weather side coating (soluble in CS_2) per 108 sq. ft.]	[15.0]	[8.0]	[15.0]	[8.0]
[Weight of reverse side coating (soluble in CS_2) per 108 sq. ft.]	....	[2.0]	....	[2.0]
Weight of weather side mineral filled coating per 108 sq. ft.	*20.0*	*10.0*	*20.0*	*10.0*
Weight of *weather side* mineral matter per 108 sq. ft. passing No. 14 (1410-micron) and retained on No. 100 (149-micron) sieve	17.0	3.0	12.0	3.0
[Weight of mineral matter per 108 sq. ft. passing No. 100 (149-micron) sieve]	[10.0]	[4.0]	[10.0]	[4.0]
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of bitumen in the coatings and the mineral matter passing No. 100 (149-micron) sieve per 108 sq. ft.	*50.0 per cent*	*15.0 per cent*	*50.0 per cent*	*15.0 per cent*
Weight of any 108 sq. ft. roll of [complete] roofing in the shipment	....	60.0	....	50.0

[a] The weight of saturant per 108 sq. ft. shall be not less than 1.6 times the weight of the dry felt.

Tentative Specifications for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 – 38 T):[4]

Section 6.—Change the values for minimum weight in the table to read as follows by the addition of the italicized figures and the omission of those in brackets:

	15-lb. Type	30-lb. Type
Minimum weight of saturated felt per 100 sq. ft. (exclusive of wrapping and packing material), lb.	14	[29] *28*

New Section.—Add a new Section 16 on Basis of Rejection to read the same as the new Section 14 recommended for addition to Specifications D 224 – 37 T, above.

Tentative Specifications for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 – 37 T):[5]

Section 6.—Change to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

(*a*) The surface *of the weather side* shall be *uniform in finish and texture.* [smooth or finely veined in appearance. Deeply ribbed surfaces shall be rejected] *The mineral surfacing shall be distributed over the entire surface (except as noted in Paragraph (b)) in a uniform layer and shall be securely embedded in the asphalt coating.*

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[4] 1939 Book of A.S.T.M. Standards, Part II, p. 1076.

[5] *Ibid.*, p. 1086.

(*b*) The coating and surfacing shall [be applied uniformly and of approximately equal thickness on both sides and up to the edges of the sheet] *cover the entire weather side of the roofing except that this side may be provided with a lapping edge approximately 2 or 3 in. in width along one edge where either the surfacing alone or both surfacing and coating may be omitted. If the edge is coated but not surfaced with mineral, a thin paper may be used to prevent sticking.*

Table I.—Change to read as prescribed in the accompanying Table II by the addition of the italicized words and figures and the omission of those in brackets.

Section 10 (*a*).—Omit the fourth sentence which reads as follows: "The ends of the rolls shall be protected with cloth covers or by other suitable means."

Section 11 (*a*).—Make the same change in this section as recommended in Section 11 (*a*) of Specifications D 224 – 37 T, above, and at the end of item (*3*) on Quantity in the revised Paragraph (*a*), add the following sentence:

Where the roofing is intended for application by the blind nailing method and is provided with a 3-in. selvage, 150 nails per "square" shall be furnished on a basis of 36-in. roofing.

Section 11 (*b*).—At the end of this paragraph add the following sentence:

Where the roofing is intended for application by the blind nailing method, there shall be furnished 2 pt. of a suitable quick-setting asphaltic cement for each roll of one "square."

New Section.—Add a new Section 14 on Basis of Rejection to read the same as the new Section 14 recommended for addition to Specifications D 224 – 37 T, above.

Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 37 T):[6]

Table I.—Change to read as prescribed in the accompanying Table III by the addition of the italicized words and figures and the omission of those in brackets.

TABLE III.—PHYSICAL REQUIREMENTS OF ASPHALT ROOFING.

Width	The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.
Area	The average area of the rolls examined shall contain sufficient roofing to cover 100 sq. ft. of roof surface (108 sq. ft. with 2 in. or no bare lapping edge, or 111 sq. ft. with 3-in. lapping edge).
Pliability at 77 F. (25 C.)	At least 8 strips out of 10 shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ¾-in. radius.
Behavior on heating to 176 F. (80 C.) for 2 hr.	Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than ⅙ in. when suspended vertically.

	Max., lb.	Min., lb.
Weight per roll, net average:		
No bare edge	....	83.0
2 or 3-in. bare edge	....	81.0
Weight of dry felt per 108 sq. ft.	....	10.8
Weight of saturant (soluble in CS_2) per 108 sq. ft.[a]	....	17.3
[Weight of weather side coating (soluble in CS_2) per 108 sq. ft.]	[20.0]	[12.0]
[Weight of reverse side coating (soluble in CS_2) per 108 sq. ft.]	[5.0]	[2.0]
Weight of weather side mineral filled coating per 108 sq. ft.	*35.0*	*16.0*
Weight of mineral matter per 108 sq. ft. passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve	[40.0]	[25.0] *20.0*
[Weight of mineral matter per 108 sq. ft. passing No. 100 (149-micron) sieve]	[10.0]	[4.0]
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of the bitumen in the coatings and the mineral matter passing No. 100 (149-micron) sieve per 108 sq. ft.	*50.0 per cent*	*15.0 per cent*
Weight of any 108 sq. ft. roll of [complete] roofing in the shipment (coarse granule surfaced portion)	....	80.0

[a] The weight of saturant per 108 sq. ft. shall be not less than 1.6 times the weight of the dry felt.

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[6] 1939 Book of A.S.T.M. Standards, Part II, p. 1083.

Section 8 (*a*).—Make the same change in this section as recommended in Section 10 (*a*) of Specifications D 248 - 37 T, above.

Section 9 (*a*).—Make the same change in this section, except as indicated below, as recommended in Section 11 (*a*) of Specifications D 224 – 37 T, above. At the end of item (*3*) on Quantity in the revised Paragraph (*a*), add the following sentence:

> Where the roofing is intended for application by the blind nailing method, and is provided with a 3-in. selvage, 150 nails per "square" shall be furnished on a basis of 36-in. roofing.

Section 9 (*b*).—Make the same change in this section as recommended in Section 11 (*b*) of Specifications D 248 - 37 T, above.

New Section.—Add a new Section 12 on Basis of Rejection to read the same as the new Section 14 recommended for addition to Specifications D 224 – 37 T, above.

Tentative Specifications for Asphalt Cap Sheet Surfaced with Coarse Mineral Granules (D 371 – 37 T):[7]

Title.—Change the title to read as follows by addition of the italicized words and the omission of those in brackets:

Tentative Specifications for [Asphalt Cap Sheet] *Wide Selvage Asphalt Roofing* Surfaced with Coarse Mineral Granules

Section 7 (*c*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> The reverse side of the sheet [shall not be coated with asphalt, surfaced with mineral granules, or dusted with talc, mica, or other fine mineral matter] *may or may not be coated. If coated, it may be surfaced with a small amount of mineral matter to prevent sticking in the roll.*

Section 10 (*a*).—Make the same change in this section as recommended in

TABLE IV.—PHYSICAL REQUIREMENTS FOR ASPHALT CAP SHEET.

Width	The roofing shall be put up in rolls and shall be either 36 or 32 in. in width, as may be specified, within a permissible variation of ±¼ in.
Area	The average area of the rolls examined shall contain sufficient roofing to cover 50 sq. ft. of roof surface: for roofing 32 in. in width, 107.3 sq. ft. ± 5 per cent, and for roofing 36 in. in width, 106.8 sq. ft. ± 5 per cent.
Pliability at 77 F. (25 C.)[a]	At least 8 strips out of 10 from the granule-surfaced portion shall not crack when bent 90 deg. at a uniform speed over a rounded corner of ¾-in. radius.
Behavior on heating to 176 F. (80 C.) for 2 hr.[a]	Not more than 1.5 per cent volatile matter loss. The granular surfacing shall not slide more than ¼ in. when suspended vertically.

	45-lb. Grade		55-lb. Grade	
	Max., lb.	Min., lb.	Max., lb.	Min., lb.
Weight per roll, net average	....	42.0	....	52.0
Weight of dry felt per 108 sq. ft.	....	[7.3] *7.1*	....	10.8
Weight of saturant (soluble in CS_2) per 108 sq. ft.[b]	....	[11.0] *10.0*	....	17.5
[Weight of coating (soluble in CS_2) per 108 sq. ft.[a]]	[20]	[12]	[20]	[12]
Weight of weather side mineral filled coating per 108 sq. ft.[a]	*35.0*	*16.0*	*35.0*	*16.0*
Weight of *weather side* mineral matter [per 108 sq. ft.] passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve *per 108 sq. ft.*[a]	[40]	[25] *20.0*	[40]	[25] *20.0*
[Weight of mineral matter per 108 sq. ft. passing No. 100 (149-micron) sieve[a]]	[10]	[4]	[10]	[4]
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of the bitumen in the coatings and the mineral matter passing No. 100 (149-micron) sieve per 108 sq. ft.	*50.0 per cent*	*15.0 per cent*	*50.0 per cent*	*15.0 per cent*
Weight of any 108 sq. ft. roll of [complete] roofing in shipment	....	40.0	....	50.0

[a] Granule-surfaced portion.

[b] The weight of saturant per 108 sq. ft. shall be not less than [1.5] *1.4* times the weight of the dry felt on the 45-lb. grade, and 1.6 times the weight of the dry felt on the 55-lb. grade.

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[7] 1939 Book of A.S.T.M. Standards, Part II, p. 1080.

Section 10 (*a*) of Specifications D 248 - 37 T, above.

Table I.—Change to read as prescribed in the accompanying Table IV by the addition of the italicized words and figures and the omission of those in brackets.

New Section.—Add a new Section 13 on Basis of Rejection to read the same as the new Section 14 recommended for addition to Specifications D 224 - 37 T, above.

II. ADOPTION OF TENTATIVE STANDARDS AS STANDARDS.

The committee recommends that the following nine tentative standards, with editorial changes in two of them as indicated below, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:

Asphalt for Use in Constructing Built-Up Roof Coverings (D 312 - 39 T),

Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (D 450 - 38 T),

Bituminous Grout for Use in Waterproofing Above Ground Level (D 170 - 39 T),

Bituminous Grout for Use in Waterproofing Below Ground Level (D 171 - 39 T),

Primer for Use with Asphalt in Dampproofing and Waterproofing (D 41 - 39 T),

Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (D 43 - 37 T), and

Asphalt Mastic for Use in Waterproofing (Asphalt Cement, Mineral Filler, Mineral Aggregate) (D 491 - 39 T),[8] with an editorial change as follows:

Section 3.—Change the sixth requirement in the tabulation to read as follows by the addition of the italicized word and the omission of the words in brackets:

[Bitumen soluble] *Solubility* in carbon disulfide.

Tentative Methods of:

Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 37 T), and

Testing Bituminous Mastics, Grouts, and Like Mixtures (D 147 - 39 T),[9] with an editorial change as follows:

Section 3 (b).—In the first sentence after the phrase "incandescent carbon filament bulb," add the phrase "or other type of electric heater of correct capacity."

III. ADOPTION AS STANDARD OF REVISIONS OF STANDARD

Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 - 27):[10]

The committee recommends that the revision of Specifications D 227 published as tentative in 1939[11] be approved for reference to letter ballot of the Society for adoption as standard. In addition to the existing revision, the committee recommends, in order to bring this standard into accord with other specifications as revised in this report, that a new Section 13 on Basis of Rejection be added to read as follows:

> 13. *Basis of Rejection*.—Failure to conform to any one of the requirements prescribed in these specifications shall constitute grounds for rejection. In case of rejection, the seller shall have the right to reinspect the rejected shipment and resubmit the lot after removal of those packages not conforming to the specified requirements.

Since the above revision is not germane to the tentative revision being recommended for adoption, the committee accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society.

IV. TENTATIVE STANDARDS AND TENTATIVE REVISION OF STANDARD CONTINUED AS TENTATIVE

Committee D-8 recommends that the following tentative standards be continued as tentative, without revision except for an editorial change in one method as given below, since these specifications and methods of test are now being reviewed and a number of revisions will probably be recommended next year:

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[8] 1939 Book of A.S.T.M. Standards, Part II, p. 1070.

[9] *Ibid.*, p. 1094.

[10] 1939 Book of A.S.T.M. Standards, Part II, p. 482.

[11] 1939 Book of A.S.T.M. Standards, Part II, p. 1182, or 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 345.

Tentative Specifications for Asphalt for Dampproofing and Waterproofing (D 449 - 37 T),

Tentative Methods of Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 - 38 T),

Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 - 37 T), and

Tentative Recommended Practice for Accelerated Weathering Test for Bituminous Materials (D 529 - 39 T),

Tentative Methods of Testing Films Deposited from Bituminous Emulsions (D 466 - 38 T),[12] with the following editorial change made in the interest of greater accuracy:

Section 2 (b).—Change to read as follows by the addition of the italicized words and figures:

(*b*) Tiles.—Unglazed ceramic tiles,[2] *white, nonvitreous, dust pressed body with an absorption range of 15 to 18 per cent (determined in accordance with the Standard Methods of Sampling and Testing Structural Clay Tile (A.S.T.M. Designation: C 112) of the American Society for Testing Materials)* approximately 6 by 6 in. *by $\frac{3}{8}$ to $\frac{1}{2}$ in. in thickness*, or of sufficient size to accommodate the opening of the mask.

Footnote 2.—Change to read as follows by the addition of the italicized words:

[2] Suitable unglazed tile can be obtained from the Robertson Art Tile Co., Morrisville, Pa., *and from Gladding-McBean and Co., Los Angeles, Calif.*

The tentative revisions of the Standard Specifications for Asphalt-Saturated Asbestos Felt for Use in Constructing Built-Up Roofs (D 250 - 27) were accepted by the Society in 1939. Owing to the fact that further revisions are to be made in order to conform to present practice, no change in status is now recommended.

The recommendations appearing in this report have been submitted to letter

TABLE V.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 37 T)	33	1	5
Spec. for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 - 37 T)	34	0	5
Spec. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 226 - 38 T)	34	0	5
Spec. for Asphalt Roofing Surfaced with Fine Mineral Granules (D 248 - 37 T)	33	1	5
Spec. for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 37 T)	34	0	5
Spec. for Asphalt Cap Sheet Surfaced with Coarse Mineral Granules (D 371 - 37 T)	34	0	5
II. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Asphalt for Use in Constructing Built-Up Roof Coverings (D 312 - 39 T)	35	1	3
Spec. for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (D 450 - 38 T)	31	0	8
Spec. for Bituminous Grout for Use in Waterproofing Above Ground Level (D 170 - 39 T)	33	3	3
Spec. for Bituminous Grout for Use in Waterproofing Below Ground Level (D 171 - 39 T)	33	3	3
Spec. for Primer for Use with Asphalt in Dampproofing and Waterproofing (D 41 - 39 T)	33	1	5
Spec. for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (D 43 - 37 T)	29	1	9
Spec. for Asphalt Mastic for Use in Waterproofing (Asphalt Cement, Mineral Filler, Mineral Aggregate) (D 491 - 39 T), with editorial change	34	1	4
Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 37 T)	33	1	5
Testing Bituminous Mastics, Grouts, and Like Mixtures (D 147 - 39 T), with editorial change	33	0	6
III. ADOPTION AS STANDARD OF REVISIONS OF STANDARD			
Spec. for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (D 227 - 27), immediate adoption	31	0	8

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[12] 1939 Book of A.S.T.M. Standards, Part II, p. 1068.

ballot of the committee which consists of 59 voting members; 39 members returned their ballots, with the results shown in Table V.

This report has been submitted to letter ballot of the committee which consists of 59 voting members; 39 members returned their ballots, of whom 35 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. M. WEISS,
Chairman.

PRÉVOST HUBBARD,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-8 presented jointly with Committee D-4 on Road and Paving Materials to the Society through Committee E-10 on Standards new Tentative Volume Correction Table for Tar and Coal-Tar Pitch. This table was accepted by Committee E-10 for publication as tentative on August 25, 1941, and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 350, bearing the A.S.T.M. serial designation D 633 - 41 T.

APPENDIX

PROPOSED SPECIFICATIONS FOR COAL-TAR PITCH FOR STEEP BUILT-UP ROOFS, DAMPPROOFING, AND WATERPROOFING[1,2]

These are proposed specifications and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. These specifications cover coal-tar pitch suitable for use as a mopping coat and for embedding slag or gravel in the construction of steep built-up roofs with inclines of 1 to 6 in. per horizontal foot, where nailing is employed. On flat built-up roofs from a level plane to inclines up to 2 in. per horizontal foot, steep pitch is suitable for embedding and coating the top ply of felt on promenade roofs to be surfaced with quarry tile or other rigid or semirigid wearing surfaces. Steep pitch is suitable for use on vertical surfaces as a mopping coat in dampproofing, or for mopping tarred felts or tarred fabrics in the construction of membrane waterproofing.

Types

2. The coal-tar pitch covered by these specifications is of one type.

Note.—Other types of coal-tar pitch for flat built-up roofs, dampproofing, and waterproofing are covered by the Standard Specifications for Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (A.S.T.M. Designation: D 450) of the American Society for Testing Materials.[3]

Primer

3. Steep pitch usually can be applied directly to concrete surfaces without priming, but, where priming is required, the material used as a primer shall be creosote conforming to the Standard Specifications for Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (A.S.T.M. Designation: D 43) of the American Society for Testing Materials.[4]

Membrane Materials

4. (*a*) For the construction of steep built-up roofs, felts conforming to the Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 227) of the American Society for Testing Materials[5] shall be used.

(*b*) For the construction of a membrane system of waterproofing, any or all of the following felts or fabrics conforming to the specifications of the American Society for Testing Materials may be used alone or in various combinations:

Felt.—Standard Specifications for Coal-Tar Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-Up Roofs (A.S.T.M. Designation: D 227).[5]

Cotton Fabrics.—Tentative Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for

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[1] These proposed specifications are under the jurisdiction of the A.S.T.M. Committee D-8 on Bituminous Waterproofing and Roofing Materials.

[2] Published as information, June, 1941.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 131.

[4] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 134.

[5] *Ibid.*, p. 144.

Use in Waterproofing (A.S.T.M. Designation: D 173),[6] coal-tar saturant.

Properties

5. The coal-tar pitch shall be homogenous and shall conform to the requirements prescribed in Table I.

TABLE I.—REQUIREMENTS FOR COAL-TAR PITCH FOR STEEP ROOFS, DAMPPROOFING, AND WATERPROOFING.

	Min.	Max.
Specific gravity, 25/25 C. (77/77 F.)	1.20	1.35
Softening point (ring-and-ball method)	150 F. (65 C.)	170 F. (77 C.)
Total bitumen soluble in carbon disulfide, per cent	65	85
Ash, per cent	...	2.0
Distillation Test:		
Total distillate by weight, 32 to 572 F. (0 to 300 C.), per cent	...	8
Residue by weight, per cent	92	...
Flash point (Cleveland open cup)	275 F. (135 C.)	...
Penetration:		
32 F. (0 C.), 200 g., 60 sec.	4	...
77 F. (25 C.), 100 g., 5 sec.	12	35
115 F. (46 C.), 50 g., 5 sec.	...	100

Methods of Sampling and Testing

6. The coal-tar pitch shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(*a*) *Sampling.*—Tentative Methods of Sampling Bituminous Materials (A.S.T.M. Designation: D 140).[7]

(*b*) *Specific Gravity.*—Standard Method of Test for Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments (A.S.T.M. Designation: D 71).[8]

(*c*) *Softening Point.*—Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).[9]

(*d*) *Bitumen Soluble in Carbon Disulfide.*—Standard Method of Test for Determination of Bitumen (A.S.T.M. Designation: D 4).[10]

(*e*) *Ash.*—Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271.).[11]

(*f*) *Distillation.*—Standard Method of Test for Distillation of Tar Products Suitable for Road Treatment (A.S.T.M. Designation: D 20).[12]

(*g*) *Flash Point.*—Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).[8]

(*h*) *Penetration.*—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).[8]

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[6] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 305.

[7] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 343.

[8] 1939 Book of A.S.T.M. Standards, Parts II and III.

[9] 1939 Book of A.S.T.M. Standards, Part II, p. 417.

[10] *Ibid.*, p. 385.

[11] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 5.

[12] 1939 Book of A.S.T.M. Standards, Part II, p. 390.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS

Since the annual meeting in June, 1940, in Atlantic City, N. J., Committee D-9 on Electrical Insulating Materials has held two meetings; one in New York, N. Y., on November 14 and 15, 1940, and the other in Washington, D. C., on March 3 and 4, 1941. The average attendance at these two meetings was 84 members and 14 visitors. These meetings were held in conjunction with Committee D-20 on Plastics because of the close cooperation between the two committees and the large number of members who serve on both committees.

The subcommittees responsible for specific work have been very active during the year and considerable progress has been made. Several projects have reached the stage where definite recommendations have been made resulting in additions or changes in the standards for which Committee D-9 is responsible. There are also a number of other projects still under way which are mentioned briefly later in this report.

The present membership of Committee D-9 is 95; of whom 47 are classified as producers, 28 as consumers, and 20 as general interest members.

The following methods under the jurisdiction of Committee D-9 have been approved as American standards by the American Standards Association on the recommendation of Sectional Committee C59 on Electrical Insulating Materials:

Standard Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 - 39; ASA C59.1-1940),
Testing Electrical Insulating Oils (D 117 - 40; ASA C59.2-1941),
Test for Insulation Resistance of Electrical Insulating Materials (D 257 - 38; ASA C59.3-1939), and
Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 - 38; ASA C59.10-1941).

The following standards, submitted on March 3, 1941, to Sectional Committee C59, are now being studied by special reviewing committees looking toward their submittal to the American Standards Association for approval as American standards:

Standard Methods of:

Testing Laminated Tubes Used in Electrical Insulation (D 348 - 39),
Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 39), and
Testing Shellac Used for Electrical Insulation (D 411 - 40).

The Methods of Test for Impact Resistance of Electrical Insulating Materials (D 256 - 38) have now been approved by letter ballot in Sectional Committee C59 and will shortly be submitted to A.S.A. for approval as American standard.

Subsequent to the 1940 annual meeting, Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

Revisions of Tentative Methods of:

Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 - 39 T),
Testing Solid Filling and Treating Com-

pounds Used for Electrical Insulation (D 176 – 37 T),
Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 40 T).

Tentative Revisions of Standard Methods of:

Testing Molded Materials Used for Electrical Insulation (D 48 – 39), and
Testing Electrical Porcelain (D 116 – 39).

These recommendations were accepted[1] by Committee E-10 on August 28, 1940; the revised tentative methods and the tentative revisions of standards appear in the 1940 Supplement to the Book of A.S.T.M. Standards.[2]

RECOMMENDATIONS AFFECTING STANDARDS

As a result of the year's work, the committee is submitting four new tentative standards, revisions of four tentative standards, an editorial change in an existing standard, and is recommending the adoption as standard of four tentative standards. The standards affected are listed in Table I. The recommendations being presented are included in detail in Appendix I, each proposed revision being accompanied by a brief explanation of the reason for the revision.

These recommendations have been submitted to letter ballot of the committee which consists of 95 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Round Phenolic Laminated Tubing for Radio Applications	25	0	41
Test for Punching Quality of Phenolic Laminated Sheets	29	0	33
Preconditioning Plastics and Electrical Insulating Materials for Testing	35	0	27
Test for Volatile Matter Content of Vulcanized Fiber	22	0	44
II. REVISIONS OF TENTATIVE STANDARDS			
Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 39 T)	47	0	19
Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 40 T)	30	0	32
Testing Glass Spool Insulators (D 550 – 39 T)	25	0	38
III. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Testing Varnishes Used for Electrical Insulation (D 115 – 39 T)	28	0	35
Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 – 38 T), as revised	25	0	41
Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 – 38 T), as revised	31	0	31
Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (D 551 – 39 T)	23	0	43
IV. EDITORIAL CHANGE IN STANDARD			
Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 – 38; ASA C59.10-1941)	30	0	33

ACTIVITIES OF SUBCOMMITTEES

In addition to the projects that have resulted in definite recommendations, there are numerous other projects on which the various subcommittees are engaged. Data are being obtained and results studied with a view to improving the present standards and developing new standards. Some of the projects that are in progress are as follows:

Subcommittee I on Insulating Var-

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-9 reported the following results of the letter ballot vote of a total of 50 ballots returned from a committee membership of 91: D 149 – 39 T, affirmative 36, negative 0, ballots marked "not voting" 14; D 176 – 37 T, affirmative 23, negative 0, ballots marked "not voting" 27; D 202 – 40 T, affirmative 25, negative 0, ballots marked "not voting" 25; D 48 – 39, affirmative 25, negative 0, ballots marked "not voting" 25; D 116 – 39, affirmative 25, negative 0, ballots marked "not voting" 25.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 257, 242, 264, 563, and 569, respectively.

nishes, Paints, and Lacquer (C. F. Hanson, chairman).—Eleven years ago work was started to develop a test method for determining the resistance of varnish to acid and alkali. A great amount of research was done at that time over a period of two or three years. For a few years following, the work was held in abeyance, and about three years ago the investigation was resumed and the scope was expanded to include resistance to sea water. One round-robin test has been completed, and the second one is in progress with five laboratories participating. The results to date indicate that a number of precautions will have to be taken in the preparation of the test specimen. Considerably more work will have to be done to eliminate these irregularities.

There is great need in the industry for a test to determine the impregnating qualities of a varnish used for impregnating electric coils. This work has been in progress since 1932. The apparatus originally designed for determining the penetration, internal drying, and bonding strength of a saturant varnish has been temporarily abandoned and a more simplified approach has been taken. A round-robin test is under way with six laboratories participating.

It has been found that the moisture content of a specimen of shellac has considerable influence upon the flow of the specimen. The conditioning of the sample so that it will always have approximately the same moisture content is therefore very desirable. To bring about this condition is not a simple matter. It has been found that the necessary conditioning is influenced by the past history of the sample. This subject has become a research project of the subcommittee.

Among the factors which influence the drying rate of a varnish film are temperature, light, percentage of relative humidity, and dissipation of solvent vapor. To control these factors for test purposes it is necessary to provide a suitable drying cabinet. This subcommittee, in cooperation with Subcommittee X on Conditioning, has completed a survey among oven manufacturers. It is expected that recommendations for suitable equipment can soon be made.

Subcommittee II on Molded Insulating Materials (W. A. Evans, chairman).—Results of tests on tensile strength specimens molded under like conditions in molds supplied by four different laboratories have been presented. Each mold had different clearances between the cavities and their corresponding faces. A mold design is to be prepared and submitted to letter ballot.[3]

Work on conditioning test specimens of plastics is being continued in cooperation with Subcommittee X on Conditioning.

It has been agreed to redraft the Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 39; ASA C59.1 – 1940) into a more simplified form with rearrangement of the test procedures covered.[3]

A new section has been appointed to formulate specifications for thermosetting molding compounds. Although the section has not had time to do any material work, it has discussed a proposed form and the grades of material to be specified. It was suggested that general specifications be written which would include a table of characteristics and values. The specifications should cover the following divisions: general purpose, high impact, heat resistance, and possibly low loss electrical grades. Mr. J. B. Lunsford of the U. S. Navy, Bureau of Ships, stressed the greater use of high impact material by the Navy.

It has been agreed to have the section

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[3] See Editorial Note, p. 396.

on Impact Fatigue take under consideration high-velocity impact tests.

Subcommittee III on Plates, Tubes and Rods (Dean Harvey, chairman).—Methods of conditioning specimens of sheets, rods, and tubes to insure reproducibility of the tests for dielectric strength, power factor, and insulation resistance are being prepared.

A Method of Test for Punching Quality of Phenolic Laminated Sheets is being submitted for publication as tentative as appended hereto.[4] This method has been prepared after a long program of cooperative tests, including the development of a standard punching die and a point rating system for evaluating the edges, surfaces, and holes in the punched test specimen. A control test also is given, consisting of a Rockwell hardness test at room temperature or at elevated temperatures, according to the temperature used in punching. These hardness tests have been found to give an indication of punching quality. The development work and the supporting data for the test for punching quality are given in Appendix II to this report.

Cooperative work with the Navy Department has resulted in the preparation of proposed Methods of Test for Product Uniformity of Phenolic Laminated Sheets which contains a group of tests that will serve to check the uniformity of the properties of a particular type of phenolic laminated material from one manufacturer. This method is published as information as appended hereto.[5]

The Specifications for Round Phenolic Laminated Tubing for Radio Applications[6] and the Method of Test for Volatile Matter Content of Vulcanized Fiber,[7] being recommended as tentative as appended hereto, were prepared by two sections of this subcommittee.

Ten of the standard methods of test under the jurisdiction of the subcommittee have been referred to members for review and preparation of a statement on the significance of these tests.

Further tests have been made to determine the ply adhesion of vulcanized fiber, and slight modifications in the equipment are being considered, in order that more consistent results may be obtained.

Standard methods of measurement of dimensions are being prepared for round, stiff tubing to insure consistent results. Studies are being made to develop a method of test for the dielectric strength of phenolic laminated tubing parallel to laminations.

J. B. Lunsford, of the Navy Department, gave a very interesting discussion at the spring meeting on the need for standard methods of test. Short-time tests are particularly desired by the Navy Department which will indicate how the materials will perform in service. The Navy Department Specification for Fish Paper for Electrical Insulation (17I11b) was reviewed with representatives of the Navy Department and various changes were suggested to bring it in line with commercial practice.

A study is being made of the method of test for flexural strength of sheet insulating material in the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 39) to determine the effect of various factors upon the accuracy of the test.

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman).—Work is continuing on the cooperative samples for neutralization number; however, several of the members have not finished their tests. It is expected that the work will be completed before the annual meeting.

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[4] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 359.

[5] Subsequent to the annual meeting this method was presented to the Society for publication as tentative, see Editorial Note, p. 396.

[6] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 347.

[7] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 352.

Two samples, one new oil and the other a used oil, have been sent out to members of the Section on Sludge Test. Each sample is to be tested in accordance with two test procedures, the sludge accumulation test and the high-pressure oxidation test, as given in the Proposed Methods of Test for Sludge Formation in Mineral Transformer Oil which are being published as information in Appendix III. It is hoped that many coöperators will make a third test of a 50:50 volumetric blend of the two samples. Attention is called to the limitations of the test and the fact that as yet there is no coordination of the test with actual operating conditions.

The Methods of Testing Electrical Insulating Oils (D 117 - 40; ASA C 59.2 - 1941) have been reviewed in their entirety to determine which sections apply to synthetic insulating liquids. In the case of these sections that do not apply to synthetic liquids, new descriptions are being written. When this work is completed, it is felt that the tests for synthetic insulating liquids should be separated from the tests for mineral insulating oils.

The Section on Saponification is coöperating with Committee D-2 on Petroleum Products and Lubricants in round-robin tests. Committee D-2 is recommending this year a revision of the Tentative Methods of Test for Saponification Number (D 94 - 39 T) which methods are under the joint jurisdiction of Committees D-2 and D-9. Committee D-9 will probably concur in this recommendation.

A more reliable vacuum gage has been designed for determining the gas content in oils and more data are to be obtained.

The work on dielectric strength testing of oil is being continued. This investigation includes a study of momentary discharges, method for high viscosity oils, rate of applying voltage, shape of electrodes, and handling of the sample. Tests for dielectric strength of synthetic insulating liquids are also being studied.

The articles on the significance of the various tests in Methods D 117 are being held in abeyance until the Section on Scope has completed its work.

An investigation is being made of the various methods now available for determining moisture in insulating oil.

Several generic names have been suggested for synthetic insulating liquids. However, a search of the Patent Office will have to be made for prior use of any of these names. It is understood that the National Electrical Manufacturers Association has a name for these compounds and a request of this Association will be made for an exact definition.

Subcommittee V on Ceramic Products (Porcelain, Glass, Etc.) (L. E. Barringer, chairman).—A round-robin series of tests is in progress in connection with the study of methods of testing steatite for mechanical strength. Preliminary results indicate satisfactory agreement for flexural strength, but additional data are required for compressive strength and resistance to impact.

Samples of five widely different types of glass were circulated among five laboratories to help in correlating the measuring technique of power factor and dielectric constant of glass over the range of frequencies for which equipment was available. The data thus accumulated have been assembled and are presented in a paper by Philip A. Richards, "Report on Round-Robin Tests of Power Factor and Dielectric Constant for Glass" which appears as Appendix VI to this report.

Work in connection with reconciling the A.S.T.M. Tentative Method of Testing Pin-Type, Lime Glass Insulators (D

468 - 39 T) and the American Standard for Insulator Tests (AIEE No. 41 - 1930; ASA C29a-1930) has been carried forward. A group has been appointed to investigate ways and means of collecting data to correlate service with tests of lime glass insulators made in accordance with the A.S.T.M. test method.

Subcommittee VI on Solid Filling, and Treating Compound (R. H. Titley, chairman).—Studies are being made of a method for determining the solubility of treating compounds in oil.

Subcommittee VII on Electrical Tests (J. A. Scott, chairman).—A draft of a new test method for thermal conductivity which has been written by Committee C-16 on Thermal Insulating Materials, was presented to the section. The section did not feel that this proposed method would be suitable for use by Committee D-9 and decided to prepare a method covering electrical insulating materials to take the place of the present Tentative Method of Test for Comparing Thermal Conductivities of Solid Electrical Insulating Materials (D 325 - 31 T).

The results of round-robin tests on solid dielectrics in connection with the measurement of power factor and dielectric constant at ultra high frequencies have resulted in a revision of the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 39 T) given in Appendix I. The results of the tests using the reactance (susceptance) variation method and four forms of apparatus have been summarized by K. G. Coutlee, R. F. Field, E. O. Hausmann, Thomas Hazen, and H. R. Meahl and are presented in a paper on "Measurements of Power Factor and Dielectric Constant at Ultra High Frequencies" in Appendix V to this report.

A request has been made to reopen activity on the question of oil power factor measurements which had been discontinued following the report of the round-robin tests. It was pointed out that from this round-robin series certain useful recommendations on electrodes and cells should be prepared and given a place in the methods.

Subcommittee VIII on Insulating Papers and Fabrics (*Treated and Untreated*) (R. W. Chadbourn, chairman).—In line with the policy of Committee D-9 to consider jointly with other organizations test methods and specifications of mutual interest, the subcommittee obtained the consent of Committee D-6 on Paper and Paper Products to the appointment of a small joint committee to study the pH method for acidity determination. The work of this group began with the consideration of the method developed by M. S. Kantrowitz and previously under consideration by Committee D-6. Possible improvements in this method, involving such things as preparation of sample, are to be considered, and a series of tests is being undertaken to determine its applicability to condenser paper.

The Paper Division is following closely the review by Committee D-6 of methods of mechanical tests for papers. Further progress has been made by this committee during the year on many methods including tensile strength, porosity, conditioning, ash, and tearing strength. A new type of edge tear tester, having a tearing edge shaped in the form of a widely spread "V," has been developed and is receiving consideration. As a result of a request received during the year for a wet tensile strength method, a simple and ingenious testing device has been developed for this purpose. Its applicability will be investigated in a set of round-robin tests.

The round-robin tests involving three types of micrometers (standard machinists', machinists' with adjusted pressure, and dead weight dial) have been completed. The results indicate that the dead-weight type is equal to the machinists' in accuracy. An alternative method of thickness determination, involving the dial gage, will therefore be prepared for inclusion in the Tentative Methods of Test for Thickness of Solid Electrical Insulation (D 374 - 36 T).[3]

During the year a section was organized to develop statements on the significance of the various tests included in the Tentative Methods of Sampling and Testing Treated Paper Used in Electrical Insulation (D 202 - 40 T).

The tests by four utility company laboratories to determine the elongation characteristics of several special samples of varnished cloth tape under conditions simulating actual use are nearing completion. A technique was developed which permitted the measurement for the first time of actual elongation of samples wound on special mandrels by field men such as cable splicers. The data obtained in these tests are expected to be of value, not only in the revision of elongation and dielectric strength requirements, but also in establishing the most desirable constructional qualifications for a tape of this type.

During the year replies were received from a number of large utilities to whom a questionnaire was sent requesting data on their use of tape and their reactions to A.S.T.M. test methods and specifications. The replies received were summarized and analyzed, and the information so obtained is expected to be of great value in further revision of the Tentative Specifications for Black Bias-Cut Varnished Cloth Used for Electrical Insulation (D 373 - 39 T) and the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 38 T).

The subcommittee has begun a coöperative study with the Underwriters' Laboratories, Inc., of the latter's proposed specifications for varnished tubing in an effort to bring the Underwriters' and A.S.T.M. specifications into closer agreement.

During the year a new section was formed for the development of test standards and specifications for treated glass fabrics. Several existing test methods for similar fabrics have already been approved for treated glass, and there are under way studies of a number of other properties such as breaking strength, weight, temperature life tests, and moisture absorption.

Subcommittee IX on Mica Products (M. P. Davis, chairman).—The grading chart for mica in the Standard Methods of Test for Grading and Classification of Natural Mica (D 351 - 38) is to be revised, and it has been recommended that a separate grading chart be prepared to cover the smaller sizes of mica films required primarily by the radio tube manufacturer.

Details of a mercury electrode to be used for making power factor and dielectric constant measurements at room temperatures are being prepared for inclusion in the Tentative Method of Test for Power Factor and Dielectric Constant of Natural Mica (D 351 - 39 T).

The preparation of color prints, transparencies, and black and white photographs for classification of mica is being continued.

The Navy Department has issued tentatively a new specification for mica which has been reviewed by members of the subcommittee.

Subcommittee X on Conditioning (Robert Burns, chairman).—The subcommittee has approved the definition for "dry room conditions" as 15 per cent relative humidity at 85 F. and for "moist room conditions" as 75 per cent relative humidity at 77 F. It has also approved the establishment of a standard laboratory atmosphere consisting of 50 ± 2 per cent relative humidity at 77 ± 2 F. The purpose of a standard laboratory atmosphere is to provide standardized controlled atmospheric influences for those rooms or chambers where testing machines are located, thereby facilitating a greater degree of reproducibility in test results.

In connection with the project of developing a standard conditioning chamber, several chambers have been supplied for various laboratory and industrial uses and performance charts from one of the humidity chambers have been examined by the members of the subcommittee.

In cooperation with Committee D-20 on Plastics, proposed Tentative Methods of Preconditioning Plastics and Electrical Insulating Materials for Testing[8] have been prepared and submitted to letter ballot of both committees.

Special Subcommittee on Significance of Tests (Dean Harvey, chairman).—This subcommittee acts as a clearing house for the material prepared by the various subcommittees and it consolidates this material wherever possible to avoid unnecessary repetition. It was suggested that a uniform procedure be established which will involve the insertion in each standard and tentative method of a brief statement regarding the significance of the test or test procedures covered, and the preparation of a more detailed discussion for separate publication.

Consideration has been given to the publication of the various articles on significance of tests so that each will have a designating number to which reference can be made in the various methods of test under the jurisdiction of Committee D-9.

This report has been submitted to letter ballot of the committee which consists of 95 voting members; 66 members returned their ballots, of whom 59 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. SMITH TAYLOR,
Chairman.

E. J. RUTAN,
Secretary.

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[8] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 320.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-9 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Specifications for:
  Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D 647 - 41 T).
New Tentative Methods of:
  Test for Product Uniformity of Phenolic Laminated Sheets (D 634 - 41 T),
Revisions of Tentative Methods of:
  Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 40 T),
  Test for Thickness of Solid Electrical Insulation (D 374 - 36 T),
Tentative Revisions of Standard Methods of:
  Testing Molded Materials Used for Electrical Insulation (D 48 - 39), in the form of the following five new tentative methods:
  Testing Molded Materials Used for Electrical Insulation (D 48 - 41 T),

Test for Distortion Under Heat of Molded Electrical Insulating Materials (D 648 – 41 T),

Test for Compressive Strength of Electrical Insulating Materials (D 649 – 41 T),

Test for Flexural Strength of Electrical Insulating Materials (D 650 – 41 T),

Test for Tensile Strength of Electrical Insulating Materials (D 651 – 41 T),

Testing Shellac Used for Electrical Insulation (D 411 – 40), and

Test for Impact Resistance of Electrical Insulating Materials (D 256 – 38), in the form of new Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 41 T).

These recommendations were accepted by Committee E-10 on August 25, 1941, and assigned the A.S.T.M. serial designations indicated above. The new and revised tentative standards as well as the tentative revisions appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 317, 354, 406, 368, 322, 334, 332, 336, 345, 629, 339.

On October 22, 1941, Committee E-10 accepted for publication new Tentative Methods of Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps, which bears the A.S.T.M. serial designation D 652 – 41 T and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 426.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentative standards covering electrical insulating materials which are referred to earlier in this report.[1] In connection with each title is given the reference to the publication in which the standard appears in its present form.

NEW TENTATIVE STANDARDS

Tentative Specifications for Round Phenolic Laminated Tubing for Radio Applications:[2]

These specifications cover round phenolic laminated tubing for radio and similar applications where high frequencies are involved. They were prepared at the request of the Radio Manufacturers' Assn.

Tentative Method of Test for Punching Quality of Phenolic Laminated Sheets:[3]

The development of this method of test is described in a report on "An Investigation of Punching Quality of Laminated Phenolic Sheet," prepared by Section H on Punching Quality of Subcommittee III, which appears in Appendix II.[4]

Tentative Methods of Preconditioning Plastics and Electrical Insulating Materials for Testing:[5]

This method has been prepared in cooperation with Committee D-20 on Plastics.

Tentative Method of Test for Volatile Matter Content of Vulcanized Fiber:[6]

This method of test was developed to obtain more consistent results and to reduce the time required to determine the amount of moisture and other volatile ingredients in vulcanized fiber of all grades and thicknesses.

REVISION OF TENTATIVE STANDARD

Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 39 T):[7]

This revision provides for the measurement of power factor at frequencies above 1 megacycle using a susceptance variation method and better indexing of the present material.

Section 1.—Change from its present form to read as follows:

1. These methods provide procedures for the determination of the power factor and dielectric constant of solid and fluid electrical insulating materials at frequencies from 25 cycles per second to 100 megacycles per second. These procedures include five bridge arrangements, and

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[1] See p. 390.

[2] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 347.

[3] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 359.

[4] See p. 403.

[5] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 320.

[6] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 352.

[7] 1939 Book of A.S.T.M. Standards, Part III, p. 745.

three resonant-circuit methods. The following tabulation gives ranges of frequencies for which the various procedures are generally deemed applicable; these ranges overlap to a considerable extent and the suitability indicated for one procedure is therefore not to be interpreted as excluding other procedures at a given frequency:

Method	Reference		Usual Frequency Range, cycles per second
	Section	Figure	
Conjugate Schering Bridge	22	7	25 to 5 000 000
High-Voltage Schering Bridge	23	8	25 to 10 000
Parallel - Resistance Bridge	24	9	1 000 to 100 000
Series-Resistance Bridge	25	10	200 to 100 000
Transformer Bridge	26	11 to 12	25 to 1 000
Resonant-Circuit, Resistance Variation	27	13	100 000 to 1 000 000
Resonant-Circuit, Susceptance Variation	28	14	10 000 to 100 000 000

Section 11.—Add a note following the first paragraph of this section to read as follows:

NOTE.—The following specifications and methods of the American Society for Testing Materials now contain special requirements covering power factor tests in addition to those described in these methods:

Tentative Specifications for Phenolic Laminated Sheet for Radio Applications (A.S.T.M. Designation: D 467),[8]

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 295),[9] and

Tentative Method of Test for Power Factor and Dielectric Constant of Natural Mica (A.S.T.M. Designation: D 351).[10]

Change Paragraph (*a*) from its present form to read as follows:

(*a*) *Sheets and Plates.*—For testing sheet materials and molded or ceramic materials in plate form at frequencies up to 1.0 megacycle per second, the test specimen shall be, preferably, either a disk 6 in. (15.24 cm.) in diameter, or a square plate 6 in. (15.24 cm.) on a side. Preferably, the thickness shall be not less than 0.1 in. (0.254 cm.) nor more than 0.3 in. (0.762 cm.). Other thicknesses may be used where it is desired to test materials of the various thicknesses in commercial use. In any case, the capacitance of the specimen shall be, preferably, not less than 100 $\mu\mu$f. When using the resonant-circuit resistance variation method, stray capacitances become troublesome and influence adversely the precision of measurement when the capacitance of the specimen is much less than 100 $\mu\mu$f.

For tests made at frequencies from 1.0 to 100 megacycles per second, the test specimen shall be, preferably, in the form of a disk 2 in. (5.08 cm.) in diameter or otherwise of the same diameter as the electrodes of the capacitor which holds the specimen. Material in commercial use will determine the thickness of the specimen under test. In any case, the capacitance of the specimen shall be, preferably, not less than 15 $\mu\mu$f. nor more than 45 $\mu\mu$f. although satisfactory results may be obtained with specimen capacitances up to 80 $\mu\mu$f.

For all tests, the thickness of the specimen shall be uniform within plus or minus 5 per cent of the nominal thickness and the surface shall be as smooth and free as possible from irregularities.

Section 13.—In the last sentence of the first paragraph of Paragraph (*a*) change the value "10 megacycle per second" to read "1.0 megacycles per second," and add a new sentence to read as follows:

With materials of high dielectric constant and electrodes of comparatively thin foil it may be desirable to introduce the test current to the foil through a copper ring or plate rather than by a single wire making contact at the center of the electrode.

At the end of the third paragraph add a new paragraph to read as follows:

For measurements at frequencies from 0.1 to 100 megacycles per second by the susceptance variation method, the electrodes shall be lead or tin foil 0.00025 to 0.002 in. in thickness. The foil electrodes shall be applied with a very thin film of petrolatum and carefully rolled down on the surface of the specimen with a narrow roller until no visible imprint can be made on the foil with the roller. Preferably, the two electrodes shall be circular and shall cover the full area of both sides of the test specimen. One satisfactory method of applying such electrodes is to start with foil larger than the specimen and trim it to size with a razor blade after it has been rolled on the specimen.

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[8] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 233.

[9] 1939 Book of A.S.T.M. Standards, Part III, p. 677.

[10] *Ibid.*, p. 800.

Section 14 (*a*).—Add the following two new sentences at the end of this paragraph:

> With the susceptance variation method it is usually necessary to place all the apparatus, exclusive of the oscillator and sometimes the vacuum-tube voltmeter, in the temperature chamber. The maximum temperature at which measurements may be made is limited by the type of adhesive used to affix the electrodes.

Section 15.—Change from its present form to read as follows:

> 15. The frequency ranges for which the following methods are, at present, deemed suitable, have been indicated in Section 1. As indicated there the ranges overlap for the several methods and the choice of method in any given situation will usually be determined by the laboratory equipment available, such as resistors, capacitors, detectors, amplifiers, insulating transformers, and the like. All procedures described in these methods are capable of yielding results of the stated accuracy.

Section 19 (*a*).—Change the last three sentences to read as follows by the addition of the italicized words and the omission of those in brackets:

> The procedure contemplated in these cases is first to secure a reading with the connections as shown, and then to [disconnect and "ground" the "high" plate of the test capacitor] *remove from the circuit the specimen capacitor, that is,* C_x; following [which] *this* the circuit is restored *to its previous condition* by changing C_2 and varying the means for changing power factor, the rest of the bridge or resonant circuit remaining unchanged. The effects of various residual errors in other parts of the circuit are thereby minimized. The equivalent parallel capacitance C_p of the test specimen will then be calculated from the change in reading of *the variable precision air capacitor,* C_2.

Section 23 (*a*).—Change the first sentence from its present form to read as follows:

> See Fig. 8. This bridge is useful where the voltage required across the specimen capacitor is in excess of about 100 v., and is applicable for all frequencies from 25 cycles per second up to about 10,000 cycles per second, the chief limitations at the higher frequencies being the amplifier used with the detector and the elimination or evaluation of stray capacitance in the resistor R_3. When C_1 can be made adjustable so that the arms R_3 and R_4 can be identical resistors the method is applicable to frequencies up to 1 megacycle per second.

Section 27.—Change the second sentence of Paragraph (*a*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> This method is [recommended] ***applicable*** for frequencies from about 100 kilocycles per second [upward] ***up to 1.0 megacycle per second.***

In Paragraph (*b*) omit the third sentence which read as follows:

> The voltmeter has been found more satisfactory at frequencies of the order of 5 megacycles per second and upward.

At the end of Paragraph (*b*) add a footnote reference to the word "source" and add a new footnote to read as follows:

> [a] The series inductance and resistance of the leads in the circuit of C_x from the junction with the circuit of C_2 may produce serious errors. The leads should be kept as short as possible and form as small a loop as possible. If several values of capacitance, which have been otherwise standardized, can be inserted in the place of the specimen capacitor, C_x, with no other change in the configuration of the circuit, the inductance and resistance of the leads may be determined. The inductance and equivalent resistance of the circuit of the variable standard capacitor, C_2 will sometimes require evaluation at the higher frequencies. See paper by R. F. Field and D. B. Sinclair, "A Method for Determining the Residual Inductance and Resistance of a Variable Air Condenser at Radio Frequencies," ***Proceedings***, Inst. Radio Engrs., Vol. 24, p. 255 (1936).

In Paragraphs (*a*) and (*c*) and in Fig. 13 change the designation of the capacitor from "C_1" to read "C_2," wherever it occurs. In Equations 17 and 18 and in the explanation of the terms in the equations, change "C_1 and C'_1" to read "C_2 and C'_2," respectively. These changes are suggested to bring Fig. 13 into agreement with other figures and with previous text.

New Procedure.—Add a new Section 28 and Paragraph VI to Appendix II comprising a description of the Susceptance Variation Method, renumbering the subsequent sections, figures, and paragraphs accordingly. Since this addition is rather extensive, the description designated as Section 28 and additional information are appended hereto.[11]

Section 29 (b).—Change the parenthetical phrase "(or kilocycles)" to read "(or kilocycles or megacycles)" to provide for reporting results of the new Susceptance Variation Method.

Bibliography.—Add the following new reference on Resonant Substitution Method:

R. F. Field and D. B. Sinclair, "A Method for Determining the Residual Inductance and Resistance of a Variable Air Condenser at Radio Frequencies," *Proceedings*, Inst. Radio Engrs., Vol. 24, p. 255 (1936).

Add a new heading "Susceptance Variation Method" and include the following new references:

L. Hartshorn and W. H. Ward, "The Measurement of the Permitivity and Power Factor of Dielectrics at Frequencies from 10^4 to 10^8 Cycles per Second," *Proceedings*, Inst. Electrical Engrs. (London), Vol. 79, p. 597 to 609 (1936).

D. B. Sinclair, "Parallel Resonance Methods for Precise Measurements of High Impedances at Radio Frequencies and a Comparison with the Ordinary Series Resonance Methods," *Proceedings*, Inst. Radio Engrs., Vol. 26, p. 1466 (1938).

K. G. Coutlee, R. F. Field, E. O. Hausmann, Thomas Hazen, and J. R. Meahl, "Measurements of Power Factor and Dielectric Constant at Ultra High Frequencies," *Preprint No. 81*, for 1940 Annual Meeting Am. Soc. Testing Mats., June 23-27.

Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 40 T):[12,13]

The withdrawal of the Standard Methods of Analysis of Roofing Felt for Fiber Composition (D 272 – 34) originally sponsored and developed by Committee D-8 on Bituminous Waterproofing and Roofing Materials was approved by the Society in 1940. Subcommittee VIII believes that the use of this method of analysis for insulating papers is too limited to justify continued reference to it in Methods D 202. It is accordingly recommended that in Methods D 202, Section 41 on Fiber Composition which refers to the analysis methods D 272 be omitted, renumbering the subsequent sections accordingly.

Committee D-6 on Paper and Paper Products prepared the Tentative Method of Test for Ash Content of Paper and Paper Products (D 586 – 40 T)[13] which was accepted by the Society at the 1940 annual meeting. It is recommended that the procedure for determining ash content as described in Methods D 202 be changed as follows:

Sections 42 to 45.—Substitute for these sections the following new Section 41, renumbering the subsequent sections accordingly:

41. The determination of ash content shall be made in accordance with the Tentative Method of Test for Ash Content of Paper and Paper Products (A.S.T.M. Designation: D 586) of the American Society for Testing Materials.[14]

Tentative Method of Testing Glass Spool Insulators (D 550 – 39 T):[15]

The following change in the mechanical strength procedure is being recommended:

Section 6.—Change the last two sentences from their present form to read as follows:

The load shall be increased from 75 per cent of the rated strength of the insulator to failure at a rate per minute of not less than 30 nor more than 60 per cent of the rated strength of the insulator. The strength of the insulator shall be considered as the load at the first sign of failure.

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[11] This revision was accepted as tentative by the Society and has been incorporated in the methods which appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 375.

[12] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 264.

[13] See also Editorial Note, p. 396.

[14] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 491.

[15] 1939 Book of A.S.T.M. Standards, Part III, p. 719.

ADOPTION OF TENTATIVE STANDARD AS STANDARD

Tentative Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 39 T):[16]

The committee recommends that these methods be approved for reference to letter ballot of the Society for adoption as standard.

Tentative Method of Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 - 38 T):[17]

The committee recommends that these methods, revised as follows, be approved for reference to letter ballot of the Society for adoption as standard:

Section 2 (c).—Change from its present form: namely,

> (*c*) *Drying Dishes.*—The drying dishes shall be thin tinfoil dishes $2\frac{1}{2}$ in. in diameter, $1\frac{1}{16}$ in. in height, and from 0.004 to 0.005 in. in wall thickness, of the type used for milk analysis and for bottle caps.

to read as follows:

> (*c*) *Drying Dishes.*—The drying dishes shall be lightweight dishes, approximately $2\frac{1}{2}$ in. in diameter and $1\frac{1}{2}$ in. in height.

Section 3 (e).—Change to read as follows by the addition of the italicized words:

> (*e*) After sieving, the sample (that portion which has passed through the *No. 40 sieve and has been retained on the No. 140 sieve*) shall be placed immediately in an airtight container to prevent absorption of moisture by the powder and the consequent error in results.

Section 4 (b) and (c).—Omit the word "tinfoil" wherever it appears in these paragraphs.

Tentative Method of Test for Arc Resistance of Solid Electrical Insulating Materials (D 495 - 38 T):[18]

The committee recommends that this method, revised as indicated below, be approved for reference to letter ballot of the Society for adoption as standard:

Section 1.—Change from its present form to read as follows:

> 1. This method of test is intended for determining the relative arc resistance of all solid electrical insulating materials. It serves to classify materials broadly rather than to bring out slight differences between materials of similar composition. Differences in arc resistance must be in the order of 2 to 1 (in seconds) in order to be significant. The general grouping of several widely used classes of insulating materials is approximately as follows:

Hot molded or laminated materials (general)	60 sec. and under
Hot molded or laminated materials (higher arc resistant types)	60 to 180 sec.
Cold molded materials (nonrefractory)	60 to 300 sec.
Cold molded materials (refractory)	240 to 480 sec.
Special materials (porcelain, wet or dry process; fused quartz; best grade slate; and similar materials)	420 to 660 sec.

Tentative Method of Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (D 551 - 39 T):[19]

The committee recommends that these methods be approved for reference to letter ballot of the Society for adoption as standard.

EDITORIAL CHANGE IN STANDARD

Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 - 38):[20]

Figure 1.—Omit the angle "20°" from the dimensions of the funnel since this angle is inconsistent with the height and diameter of the frustum of the conical funnel.

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[16] 1939 Book of A.S.T.M. Standards, Part III, p. 662.
[17] *Ibid.*, p. 695.
[18] 1939 Book of A.S.T.M. Standards, Part III, p. 736.
[19] *Ibid.*, p. 698.
[20] *Ibid.*, p. 239.

APPENDIX II

AN INVESTIGATION OF PUNCHING QUALITY OF LAMINATED PHENOLIC SHEET

REPORT OF SECTION H ON PUNCHING QUALITY, OF SUBCOMMITTEE III ON PLATES, TUBES, AND RODS

The purpose of this investigation was to find what relation exists between any of the physical and mechanical properties of laminated phenolic sheet and its punching quality, with the thought in mind that a measurement of these properties would thus provide a measure of punching quality.

In connection with this investigation, a point scoring system was developed by which it is possible to give a rating or score to the quality of the punching.

Methods of Test and Materials Studied:

Tests were made upon all properties which it was thought would have any direct relation to punching quality. These tests included 24-hr. water absorption, Izod impact, cold flow, cold Rockwell hardness, and hot Rockwell hardness taken at two different temperatures. All of the tests were made in accordance with the standard A.S.T.M. methods with the exception of the hot Rockwell hardness and cold flow described below.

These tests were conducted on grades XP, XXP, XXXP, X, XXX and CE in thicknesses ranging from $\frac{1}{32}$ to $\frac{1}{8}$ in. The following companies collaborated in this work:

Westinghouse Electric & Manufacturing Co.,
The Formica Co.,
Western Electric Co.,
National Vulcanized Fibre Co.,
Continental-Diamond Fibre Co., and
Synthane Corp.

Considerable credit should go to G. H. Mains, of the National Vulcanized Fibre Co., for his excellent work in pioneering and coordinating the work of this committee and in carrying on this work along with the chairmanship of several other committees.

To evaluate punching quality properly, it was felt necessary to construct a special punch and die which would include round holes ranging in size from $\frac{1}{16}$ up to $\frac{1}{4}$ in. in diameter, the largest hole having its edge 0.045 in. from the edge of the piece. A rectangular hole was placed in the center to check the effect of small radius ($\frac{1}{32}$ in.) corners on the surface sheet.

TABLE I.—PUNCH PRESS CONDITIONS

Collaborator	Length of Stroke, in.	Speed of Punching	
		Revolutions per min.	Lineal Speed (Idle), in. per min.
Continental	2	105	210
Formica	1⅝	73	110
National	1⅞	114	202
Western Electric	1½	135	203
Westinghouse	3	90	270

The presses used by the various collaborators ranged from 25 to 30 tons; the speed of punching and length of stroke are given in Table I. The details of the punch and die are shown in Figs. 1 and 2. The size of the punchings used in the tests and the location of the holes are given in Fig. 3.

Test Data:

The point rating or grading chart mentioned above is described in the proposed Tentative Method of Test for Punching Quality of Phenolic Laminated Sheets.[1] Under this rating system, five of the collaborators made observations upon the punching quality of the various grades and thicknesses. One series of observations was made on samples punched at room temperature and another series of observations on the same set of samples punched while hot.

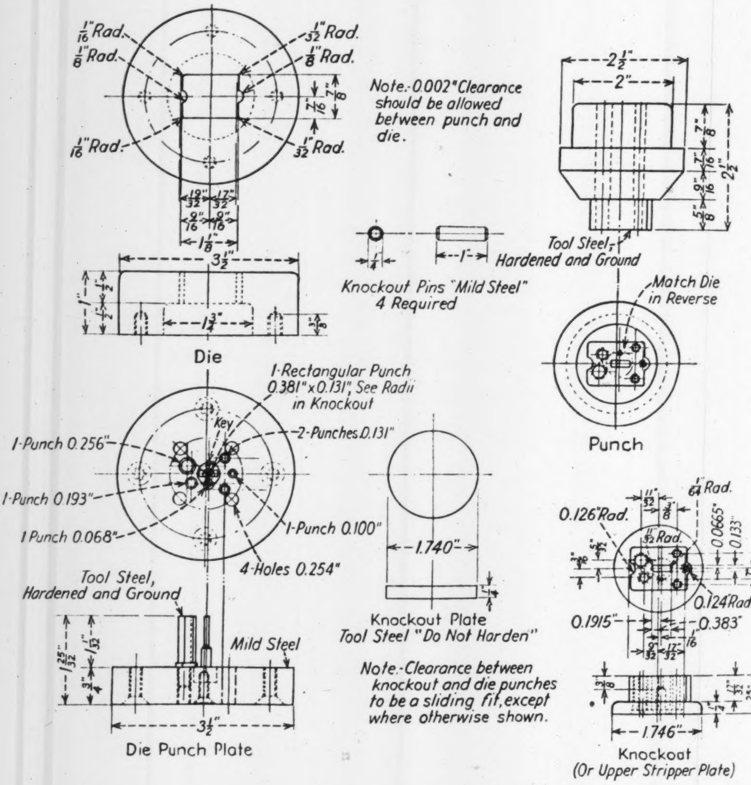
1" 16 Rad.
1" 8 Rad.
1" 16 Rad.
1" 32 Rad.
1" 8 Rad.
1" 32 Rad.
Note.-0.002" Clearance should be allowed between punch and die.
Knockout Pins "Mild Steel" 4 Required
Tool Steel, Hardened and Ground
Match Die in Reverse
Die
Punch
1-Rectangular Punch 0.381" x 0.131", See Radii in Knockout
Key
2-Punches 0.131"
1-Punch 0.256"
1-Punch 0.193"
1 Punch 0.068"
1-Punch 0.100"
4-Holes 0.254"
Tool Steel, Hardened and Ground
Mild Steel
Die Punch Plate
1.740"
Knockout Plate Tool Steel "Do Not Harden"
Note.-Clearance between knockout and die punches to be a sliding fit, except where otherwise shown.
0.126" Rad.
1" 64 Rad.
11" 32 Rad.
0.124" Rad.
0.0665"
0.133"
0.1915"
0.383"
1.746"
Knockoat (Or Upper Stripper Plate)

FIG. 1.—Details of Die and Punch.

There was, in general, fairly good agreement between the collaborators in the reports on relative punching quality of grades and thicknesses, although in some instances, one of the collaborators might be out of line on one or two grades. Data are shown on Tables II and III. In the case of grade X-101, ⅛ in. in thickness, punched cold, however, differences in rating were due to the fact that two collaborators succeeded in obtaining some punchings at least which were whole while others found the punchings to crack so badly as to be worthless.

To check the effect of rating by different observers on the same punchings, three observers rated one complete set of the punchings. The first report showed quite a variation in ratings on

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[1] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 359.

TABLE II.—SUMMARY OF PUNCHING GRADINGS BY COLLABORATORS, PUNCHING TESTS AT ROOM TEMPERATURE.

Grade	Sample	Thickness, in.	Average Point Grading on Arbitrary Scale[a]						
			Westinghouse	Formica	Western Electric	National	Continental	Average of Five Collaborators	Maximum Deviation from Average
XP (Dilecto)	No. O-365	1/16	93	93	97	92	93	94	+3
	No. O-366	3/32	83	73	87	87	83	83	−10
	No. O-367	1/8	83	63	73	77	73	74	−11
XP-208 (Phenolite)	No. 1073	1/16	86	86	97	83	93	89	+8
	No. 1074	3/32	70	80	80	80	83	77	−7
	No. 1075	1/8	50	63	57	68	70	62	−12
XXP (Dilecto)	No. O-363	1/16	90	86	97	90	90	91	+6
	No. O-364	1/8	..	63	52	70	62	62	−10
XXXP (Dilecto)	No. O-360	1/16	93	80	83	83	87	85	+8
	No. O-361	3/32	66	62	63	72	65	66	+6
	No. O-362	1/8	57	0	0	0	0	11	+46
XXX (Dilecto)	No. O-368	1/32	..	83	83	83	90	85	+5
	No. O-369	1/16	42	35	60	33	63	47	+16
	No. O-370	3/32	0	0	0	0	43	8	+35
X-101 (Phenolite)	No. 1070	1/16	80	73	87	80	83	81	−8
	No. 1071	3/32	50	63	57	75	73	64	−14
	No. 1072	1/8	0	41	0	0	48	20	+28
CEO-552A (Phenolite)	No. 1076	1/16	93	86	90	85	93	88	+5
	No. 1077	3/32	83	86	87	83	90	86	+4
	No. 1078	1/8	76	80	80	80	87	81	+6

TABLE III.—SUMMARY OF PUNCHING GRADINGS BY COLLABORATORS, STRIPS HEATED 15 MIN. AT 135 C. BEFORE PUNCHING.

Grade	Sample	Thickness, in.	Average Point Grading on Arbitrary Scale[a]						
			Westinghouse	Formica	Western Electric	National	Continental	Average of Five Collaborators	Maximum Deviation from Average
XP	No. O-365	1/16	83	96	97	83	83	88	+9
	No. O-366	3/32	76	76	73	73	77	75	±2
	No. O-367	1/8	63	56	42	50	55	53	−11
XP-208	No. 1073	1/16	83	90	97	73	87	86	−13
	No. 1074	3/32	80	83	80	65	73	76	−11
	No. 1075	1/8	63	70	57	52	63	61	±9
XXP	No. O-363	1/16	86	90	97	78	90	88	−10
	No. O-364	1/8	66	63	57	65	57	61	+5
XXXP	No. O-360	1/16	96	90	100	88	93	93	+7
	No. O-361	3/32	93	86	93	85	83	88	±5
	No. O-362	1/8	83	73	80	75	80	78	±5
XXX	No. O-368	1/32	..	93	100	93	93	95	+5
	No. O-369	1/16	83	83	73	[b]	87	82	−9
	No. O-370	3/32	66	83	70	55	73	69	±14
X-101	No. 1070	1/16	86	83	93	80	80	84	+9
	No. 1071	3/32	76	76	70	67	80	74	−7
	No. 1072	1/8	73	40	67	42	77	60	−18
CEO-552A	No. 1076	1/16	96	90	93	90	90	92	+4
	No. 1077	3/32	96	82	93	85	87	89	+7
	No. 1078	1/8	96	80	90	80	80	85	+11

[a] Average point grading for quality of edge, surface, and holes.

[b] Lost.

certain punchings. For instance, pronounced bulging around the holes was in some instances rated as a point rating against the surface, whereas it should actually apply against the holes. However, in these instances the point rating of edge, surface, and holes usually adds up to about the same total point rating. In other instances, some observers rated a surface as "good" which others rated as "fair" or as "very good." However, a second series of observations taken by three of the collaborators working together resulted in a more complete agreement as to the ratings of the various grades and thicknesses. These results are shown in Table IV.

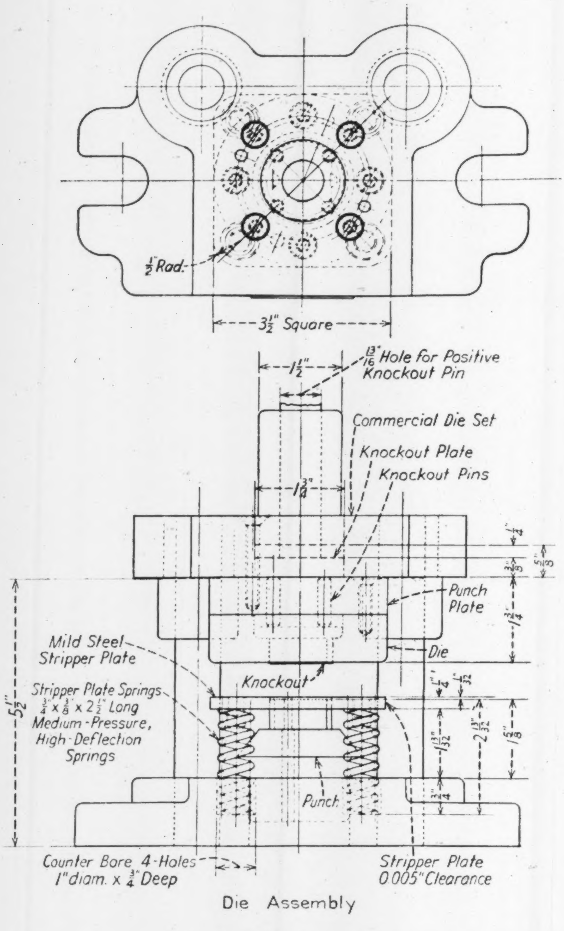

1/2" Rad.
3 1/2" Square
1 1/2"
13/16" Hole for Positive Knockout Pin
Commercial Die Set
Knockout Plate
Knockout Pins
1 3/4"
1/4"
3/8"
5/8"
Punch Plate
1 3/4"
Die
Mild Steel Stripper Plate
Knockout
Stripper Plate Springs 3/4" x 3/8" x 2 1/2" Long Medium-Pressure, High-Deflection Springs
5 1/2"
1/4"
1/32"
1 13/32"
2 13/32"
1 5/8"
3/4"
Punch
Counter Bore 4-Holes 1" diam. x 3/4" Deep
Stripper Plate 0.005" Clearance
Die Assembly

FIG. 2.—Die Assembly.

The photographs in Appendix II of the proposed method[1] indicate various point gradings of edge, surface, and holes. These photographs may serve as a guide to the proper grading of punchings.

Rockwell Hardness Tests at Room Temperature:

The Rockwell hardness tests at room temperature were conducted in accordance with the procedure outlined in Sections 39 to 42 of the Standard Methods of Testing Sheet and Plate

TABLE IV.—SUMMARY OF POINT GRADINGS IN PUNCHING TESTS.
RESULTS OF SECOND SERIES OF OBSERVATIONS BY THREE COLLABORATORS WORKING TOGETHER.

Grade			XXXP			XXP		XP			XXX			X-101			XP-208			CEO-552A		
Sample Number			O-360	O-361	O-362	O-363	O-364	O-365	O-366	O-367	O-368	O-369	O-370	1070	1071	1072	1073	1074	1075	1076	1077	1078
Thickness, in			1/16	3/32	1/8	1/16	1/8	1/16	3/32	1/8	1/32	1/16	3/32	1/16	3/32	1/8	1/16	3/32	1/8	1/16	3/32	1/8
		Laboratory																				
Cold Punching	Edge	1	95	90	..	95	90	95	90	85	95	25	..	90	85	0	95	90	85	85	80	75
		2	95	90	..	95	85	95	90	90	85	25	..	90	80	0	95	90	90	90	85	70
		3	95	90	..	95	90	95	95	90	90	25	..	80	80	0	95	95	90	90	80	70
	Surface	1	60	40	..	75	40	85	80	70	60	25	..	60	50	0	85	80	70	80	75	75
		2	55	40	..	75	40	85	80	70	60	40	..	50	50	0	70	65	50	80	75	75
		3	70	60	..	75	50	85	85	70	80	50	..	50	50	0	85	85	70	80	75	75
	Holes	1	95	80	..	95	80	95	90	75	95	75	..	90	90	0	95	90	75	95	95	90
		2	95	85	..	95	85	95	90	90	85	75	..	90	90	0	95	90	90	95	90	90
		3	95	90	..	95	90	95	95	90	100	70	..	90	90	0	95	95	90	100	95	90
	Average	1	83	72	..	90	70	92	87	77	83	42	..	80	75	0	92	87	77	85	83	80
		2	82	72	..	88	70	92	87	83	77	47	..	77	73	0	87	82	77	88	83	78
		3	87	80	..	88	77	93	93	83	90	48	..	73	73	0	93	93	83	90	83	78
Hot Punching, heated 15 min. at 135 C.	Edge	1	..	95	80	80	65	80	65	50	..	..	95	95	80	70	80	65	55	95	85	80
		2	..	85	75	80	60	80	70	50	..	..	..	80	70	60	85	70	60	95	90	90
		3	..	85	70	80	60	80	70	60	..	..	90	80	80	50	85	70	60	95	85	80
	Surface	1	..	85	70	80	65	85	80	75	..	..	40	65	40	30	80	80	75	80	75	75
		2	..	80	80	75	50	80	75	50	..	..	..	60	40	25	75	50	40	80	75	75
		3	..	85	80	75	40	80	70	50	..	..	50	60	40	25	70	50	40	85	70	70
	Holes	1	..	75	75	75	65	85	75	25	..	..	30	80	80	25	60	50	25	95	90	85
		2	..	90	80	80	70	85	80	70	..	..	..	80	75	80	85	75	60	100	95	95
		3	..	90	80	80	70	90	80	70	..	..	90	90	80	80	90	80	70	100	90	90
	Average	1	..	85	75	78	65	83	73	50	..	..	55	80	67	42	73	65	52	90	87	80
		2	..	85	78	78	60	82	75	57	..	..	..	73	62	55	82	65	53	92	87	87
		3	..	87	77	78	57	83	73	60	..	..	77	77	67	52	82	67	57	93	82	80

TABLE V.—SUMMARY OF ROCKWELL HARDNESS TESTS BY VARIOUS COLLABORATORS AT 25 C.
Note.—Numbers under collaborators names indicate order in which Rockwell hardness tests were made.

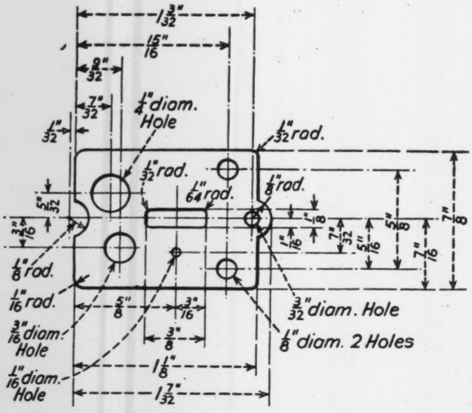
Grade	Sample	Thickness, in.	Rockwell Hardness, M Scale							
			National (1) April, 1938	Western Electric (2) (72 F., 22 C.)	Formica (3)	Westinghouse (4)	Continental (5)	National (6) March, 1939	Average	Maximum Deviation from Average
XP	No. O-365	1/16	67	70	79	83	79	84	77	−10
	No. O-366	3/32	69	74	79	83	81	79	77	−8
	No. O-367	1/8	73	71	79	84	83	78	78	−7
XP-208	No. 1073	1/16	81	82	89	89	88	84	86	−5
	No. 1074	3/32	84	74	89	84	91	86	86	−12
	No. 1075	1/8	90	76	89	91	92	85	89	−13
XXP	No. O-363	1/16	80	85	89	92	93	90	88	−8
		3/32	..	..	..	..	..	..	..	..
	No. O-364	1/8	90	80	89	90	89	85	87	−7
XXXP	No. O-360	1/16	97	101	106	105	105	103	103	−6
	No. O-361	3/32	104	103	106	109	107	108	106	±3
	No. O-362	1/8	108	107	106	109	111	109	108	+3
XXX	No. O-368	1/32	98	98	108	..	92(?)	104	100	±8
	No. O-369	1/16	106	106	108	110	112	109	109	±3
	No. O-370	3/32	110	101	108	113	113	112	110	−9
X-101	No. 1070	1/16	107	106	111	111	112	110	110	−4
	No. 1071	3/32	108	109	111	113	112	110	111	−3
	No. 1072	1/8	112	110	111	113	115	111	112	+3
CEO-552A	No. 1076	1/16	105	106	112	111	110	108	109	−4
	No. 1077	3/32	108	110	112	112	112	112	111	−3
	No. 1078	1/8	108	110	112	112	113	111	111	−3
Average of all values			96	94	100	101	101	99		

Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229 - 39).[2] A summary of the Rockwell hardness tests at room temperature made by the various collaborators is given in Table V. There are three sources of variation in the Rockwell hardness values:

1. Differences in equipment and method between the various collaborators,

2. Possible change in the Rockwell hardness values of the materials themselves due to aging or change in conditions during the period of 11 months which was required to complete the round-robin tests, and

3. Variation in Rockwell hardness value between various specimens of the same grade and thickness.



For Materials $\frac{1}{64}$" to $\frac{1}{8}$" in Thickness, Inclusive. Variations of ± 0.005" Permitted, Unless Otherwise Specified.

FIG. 3.—Punchings Used in Investigation of Punching Quality of Laminated Phenolic Sheet.

In order to eliminate the factor of age as much as possible, two checks were made by one collaborator (National Vulcanized Fibre) at the beginning of and at the completion of the round-robin tests. These showed that there was a slight increase (only 2 to 4 points) in hardness of all grades on the average during the 11-month period, except for the grade XP sample where the average increase in hardness was 11 Rockwell numbers.

For the other samples, then, a comparison of the Rockwell hardness values reported by the various laboratories gives a fairly good idea of how close such values can be checked without particular stressing of conditioning or period of aging, and upon samples which were cut from the same group of sheets molded at one time from the same batch of material and consequently include variations between sheets and between various portions of the same sheets.

Considered as a whole there is very good agreement in the Rockwell hardness values at room temperature. The average of the hardness values for four of the collaborators for all of the specimens is M-101, M-100, M-99, and M-101, respectively. The average value of M-94 for the other collaborator (Western Electric) seems to indicate that these readings are consistently low for some reason. All of the above values were taken within a 6-month period. The average value of the specimens taken several months earlier, April, 1938, was M-96 compared with M-99 in March, 1939, measured on the same specimens and machine. The value of M-92 obtained by Continental-Diamond Fibre on grade XXX, $\frac{1}{32}$ in. in thickness, seems to be in error.

Hot Rockwell Hardness Tests:

The hot Rockwell hardness tests were made at temperatures of 105 and 135 C. following the "Procedures for Rockwell Test at Elevated Temperatures" described in the proposed method.[1] The hot Rockwell hardness tests were not quite as satisfactory. This was possibly due to inexperience with this test on the part of some of the collaborators, to the directions of test being not quite specific enough, or because of some differences in the testing machines which shows up in the hot Rockwell test. The hot Rockwell hardness values obtained by the Western Electric and National Vulcanized Fibre Companies, both of whom had made previous hot Rockwell comparative tests, were fairly consistent. Values reported by Formica also agreed quite well with these, although they were made only on material $\frac{3}{32}$ in. in thickness. The other two collaborators evidently made their measurements too rapidly and too close together to obtain satisfactory curves.

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[2] 1939 Book of A.S.T.M. Standards, Part III, p. 265.

TABLE VI.—SUMMARY OF HOT ROCKWELL HARDNESS TESTS BY COLLABORATORS.

Specimens heated 15 min. at 135 C., then Rockwell hardness values plotted against time after removal from oven. Values at 60 and 120 sec. shown.

Grade	Sample	Thickness, in.	Rockwell Hardness, M Scale					
			Western Electric		National		Formica	
			60 sec.	120 sec.	60 sec.	120 sec.	60 sec.	120 sec.
XP	No. O-365	$\frac{1}{16}$	23	33	41	55	..	..
	No. O-366	$\frac{3}{32}$	26	33	35	48	26	36
	No. O-367	$\frac{1}{8}$	22	31	32	46	..	..
XP-208	No. 1073	$\frac{1}{16}$	31	37	42	54	..	..
	No. 1074	$\frac{3}{32}$	27	35	39	49	29	40
	No. 1075	$\frac{1}{8}$	25	33	32	45	..	..
XXP	No. O-363	$\frac{1}{16}$	26	35	40	53	24	39
	No. O-364	$\frac{1}{8}$	26	34	33	46	..	..
XXXP	No. O-360	$\frac{1}{16}$	31	45	46	66	..	..
	No. O-361	$\frac{3}{32}$	35	47	44	60	36	56
	No. O-362	$\frac{1}{8}$	37	53	42	64	..	..
XXX	No. O-368	$\frac{1}{32}$	61	65	87	98	..	..
	No. O-369	$\frac{1}{16}$	75	87	95	104	..	..
	No. O-370	$\frac{3}{32}$	78	92	93	108	78	92
X-101	No. 1070	$\frac{1}{16}$	89	97	96	104	..	..
	No. 1071	$\frac{3}{32}$	88	98	93	101	66	85
	No. 1072	$\frac{1}{8}$	85	92	93	102	..	..
CEO-552A	No. 1076	$\frac{1}{16}$	85	93	90	100	..	..
	No. 1077	$\frac{3}{32}$	81	92	92	100	..	104
	No. 1078	$\frac{1}{8}$	88	95	89	101	..	..

The values obtained by the three collaborators using the proposed tentative method[1] are given in Table VI. It should be noted that these hot Rockwell hardness values distinctly bring out the differences between the plasticized or punching grades and the hard grades. They also show that the hot Rockwell hardness value for the hard grades at the end of 60 sec. after removal from the oven is about the same as the cold Rockwell hardness value for the punching grades. The hot Rockwell hardness values for the softer punching grades are very low, probably too low, making the specimen too soft under these conditions for best punching.

Hot Punching Under Other Temperature Conditions:

Four of the collaborators also punched the samples under a third set of conditions, that generally used by their respective fabricating plants. These conditions are shown in Table VII.

In general, the collaborators reported no great difference between the punching test at 135 C. after heating for a 15-min. period, and their own plant hot punching condition as regards quality of the punchings. National Vulcanized Fibre reported that the lower temperature of 108 C. and the shorter time improved somewhat the punching quality of the cold-punching grades XP in the $\frac{1}{8}$-in. thickness with less drag on the edges and less bulging of holes. Of course, this temperature was too low for best results with grades XXX or

X-101. Westinghouse reported slightly better results with grades XXX and X-101 at 140 C. than at the 135 C. condition.

Comparison of Punching Properties with Other Physical Tests:

Table VIII shows a summary of all tests conducted on the various grades along with the average point score for cold and hot punchings taken from Table IV. The various tests were made in accordance with A.S.T.M. standard methods except for the hot Rockwell hardness, already described. The cold flow tests were made with the Rockwell machine, measuring the change in indentation under a 100-kg. load over a 10-min. period.[3]

TABLE VII.—ADDITIONAL SET OF HOT PUNCHING CONDITIONS USED BY VARIOUS COLLABORATORS AND REPRESENTING THEIR USUAL FABRICATING PLANT PRACTICE.[a]

Collaborator	Heating Device	Temperature	Time of Heating Strips, min.
Continental	electric oven	185 to 190 C.	¾ to 2¼[b]
Formica	steam hot plate	96 C.[c] (covered) 119 C. (uncovered)	..
National	steam hot plate	108 C.[d]	2
Westinghouse	steam heated oven	140 C.	10

[a] Western Electric reported that conditions varied so much with grade thickness and application in its plant practice that no one set of conditions could be specified as general plant practice.

[b] Time is varied according to the thickness of the strip and to the grade until the strip is flexible enough to suit the operator.

[c] Thin sheets of the punching grades are heated on the covered hot plate at 96 C., while thick sheets of punching grades and all thicknesses of grade XXX are heated at 119 C.

[d] This temperature is primarily for punching grades. Thick sheets of grade XXX are generally heated in an electric oven at 175 to 225 C. from ½ to 2 min.

Conclusions:

A study of these data by the committee has led to the following conclusions.

1. In general, there is good agreement between the various collaborators upon the punching quality of the materials punched under standard conditions using the die which was circulated. There is even closer agreement between the three observers who checked one set of the punchings. Variations that occur in rating quality of edge, surface, or holes are due to one observer attaching more importance to certain defects than did the others; in these cases the average value of the punching was, however, generally little affected.

2. The greatest factor affecting punching quality is thickness of the sheet. Any grade can be satisfactorily punched cold in thin sizes $\frac{1}{32}$ in. and under, and most grades can be satisfactorily punched hot up to and including $\frac{1}{16}$ in. in thickness. Cold punchings of grades X and XXXP in $\frac{1}{8}$-in. thickness were worthless. Cold punching of grade XXX $\frac{3}{32}$-in. thickness was also worthless.

3. There is an optimum heating condition for punching the various grades, depending upon the grade and thickness. Too high a temperature makes the material too soft which may result in nearly as poor punchings as too low a temperature.

4. Rockwell hardness seems to be definitely tied into punching quality for a definite grade of material or even for all absorbent paper base grades. For

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[3] *The method for measurement of cold flow using the Rockwell hardness machine was as follows:*

Apparatus.—Rockwell hardness machine, ¼-in. ball penetrator, and a stop watch.

Specimen.—1 by 1 in. square with pile-up of minimum number of layers to produce a total thickness of at least ¼-in.

Temperature.—25 ± 2C.

Testing.—Place the pile-up on the flat anvil, and with ¼-in. ball in place, bring minor load to bear by screwing up anvil. As soon as adjusted to zero, apply the major load of 100 kg. At the instant the crank handle mechanism comes to a stop, start the stop watch and take an instantaneous reading. Take a second reading at the end of 10 min.

Report.—The cold flow value is reported as the number of divisions change during 10 min

TABLE VIII.—SUMMARY OF RESULTS SHOWING COMPARISON OF PUNCHING PROPERTIES WITH OTHER PHYSICAL TESTS.

Grade		XXXP			XXP		XP			XXX			X-101			XP-208			CEO-552A		
Sample Number		O-360	O-361	O-362	O-363	O-364	O-365	O-366	O-367	O-368	O-369	O-370	1070	1071	1072	1073	1074	1075	1076	1077	1078
Thickness, in.		1/16	3/32	1/8	1/16	1/8	1/16	3/32	1/8	1/32	1/16	3/32	1/16	3/32	1/8	1/16	3/32	1/8	1/16	3/32	1/8
Cold Punching Grading (Average from Table IV), points		84	75	..	89	72	92	89	81	83	46	..	77	74	0	91	87	79	88	83	79
Hot Punching Grading (Average from Table IV), points		..	86	77	78	61	83	74	56	..	..	66	77	65	49	79	66	54	92	85	82
Cold Rockwell Hardness, 100 kg., 1/4-in. ball		101.5	106	109.5	89.5	86.5	79	79	75	102	108.5	112.5	109.2	108.5	109.2	81.5	81	82	108.5	109.5	110.2
Hot Rockwell Hardness at 105 C.	60 sec. 120 sec.	M-57 M-74	M-54 M-73	M-60 M-81.5	M-42 M-56	M-47 M-57	M-47 M-58	M-42.5 M-52	M-39 M-50	M-89.5 M-98	M-98.5 M-106	M-100 M-106	M-100 M-105	M-98 M-105.5	M-96.5 M-105	M-49 M-60	M-41.5 M-53.5	M-42 M-52	M-98 M-102	M-102 M-105	M-99 M-106
Hot Rockwell Hardness at 135 C.	60 sec. 120 sec.	M-46 M-66	M-43.5 M-60	M-41.5 M-64	M-40 M-53	M-33 M-46	M-41 M-55	M-35 M-47.5	M-32 M-46	M-86.5 M-97.5	M-95 M-104	M-93 M-108	M-96 M-104	M-93 M-101	M-93 M-102	M-41.5 M-54	M-39 M-49	M-32 M-45	M-89.5 M-100	M-92 M-100	M-89 M-101.5
Cold Flow, 10 min., 100 kg., 1/4-in. ball		17.3	15.8	15.6	28.1	32.6	33	32.5	36.2	9.3	9.4	8.5	9.0	9.3	9.2	33.5	31.8	36.0	10.5	10.6	10.5
Water absorption, 25 C.:																					
Volatile at 105 C., per cent		0.57	..	..	0.64	..	0.67	..	..	..	0.48	..	0.58	..	..	0.44	..	..	0.53	..	..
Water absorption, 24 hr., per cent		1.10	..	..	2.71	..	4.96	..	..	..	0.86	..	4.18	..	..	3.10	..	..	1.04	..	..
Izod Impact, ft-lb.	Machine direction	..	..	0.43	..	0.48	..	..	0.76	..	..	0.53	..	..	0.83	..	..	0.80	..	..	2.48
	Cross direction	..	..	0.40	..	0.56	..	..	0.81	..	..	0.47	..	..	0.75	..	..	0.79	..	..	2.17

paper base punchings the data indicate that Rockwell hardness values must be between M-65 and M-85 for good cold punchings with the standard die in $\frac{3}{32}$ in. or greater thicknesses. For materials harder than this, heating to such a temperature that the hot Rockwell value at 120 sec. lies in this range, M-65 to M-85 will also give good punchings. Material $\frac{1}{16}$ in. in thickness can be satisfactorily punched cold up to M-105.

Rockwell hardness is particularly tied in with the condition of the edge and of the hole but bears no direct relation to the condition of the surface which depends largely upon the resin content of the surface sheet or ply and the bond between it and the other plies.

5. Water absorption, impact strength, and cold flow have little direct relation to punching quality except perhaps for some certain grade. Impact strength is, of course, of great importance in the ability of a punching to withstand riveting or other severe assembling operations.

In view of these conclusions a test method was prepared covering the standard die, standard punching conditions, and standard grading system substantially as used in this study, as a referee method for determining punching quality.

Supplementing the referee method, a second method was prepared as a control test, covering the determinations of Rockwell hardness both at room temperatures and at elevated temperatures.

The proposed Tentative Method of Test for Punching Quality of Phenolic Laminated Sheets[1] is offered as a result of these investigations.

Respectfully submitted on behalf of Section H on Punching Quality,

S. W. PLACE, *Chairman.*
G. H. MAINS
E. O. HAUSMANN

APPENDIX III

PROPOSED METHODS OF TEST FOR SLUDGE FORMATION IN MINERAL TRANSFORMER OIL[1]

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

INTRODUCTION

Investigations under the direction of Subcommittee IV on Liquid Insulation of Committee D-9 on Electrical Insulating Materials have shown that the results obtained with either of these methods of test are reproducible if proper care be exercised by the test operator. It must be clearly recognized that to get reproducible results the test operator must closely follow the procedure described making certain that the conditions of the oxidation are carefully maintained within the prescribed limits. Variation from the procedure given will frequently give widely divergent test results.

These proposed test procedures are published in order to obtain comment and criticism. No data are available which correlate the results obtained with the commercial use of transformer oil. In publishing the two procedures for test, no implication, direct or implied, is made that either procedure is of commercial operating value. From the comments and criticism obtained as a result of the publication of these test procedures, together with the results of the continued study of the test methods by the members of Subcommittee IV, Committee D-9, it is hoped that a true evaluation of their commercial importance may be obtained.

For fundamental details describing each of the test procedures outlined, reference should be made to the following papers: F. M. Clark, and E. A. Snyder, "Testing for Sludge Formation in Mineral Transformer Oil,"[2] and F. M. Clark, "Studies in the Oxidation of Mineral Transformer Oil."[2]

In a general sense, Method A, the Sludge Accumulation Test, is considered of value in studying the sludge forming propensity of a new grade of mineral transformer oil before commercial application. Method B, the High-Pressure Oxidation Test, is considered of value in checking the sludge forming characteristics of a standard grade of oil in order to control the continuity of this property from shipment to shipment. Which type of test is best adapted for determining the condition of mineral oil in transformer use is a decision which must await the results of further study.

1. *Scope.*—These methods of test are applicable in the evaluation of the sludge forming tendency in new, mineral transformer oils. Two methods are covered as follows:

(*a*) *Method A*, the sludge accumulation test, is applicable to new, mineral transformer oils except those that have been "stabilized" by the addition of retarders, stabilizers, and negative catalysts.

(*b*) *Method B*, the high-pressure oxidation test, is applicable to new, mineral transformer oils except the so-called highly refined, white oils of zero iodine value, and mineral oil containing retarders, stabilizers, and negative catalysts.

Method A: Sludge Accumulation Test

2. *Nature of Test.*—The sludge accumulation test for mineral transformer oil evaluates the propensity of an oil to deposit an insoluble sludge under definitely prescribed conditions of oxidation in the presence of a copper catalyst for 72, 168, and 336 hr. The total evaluation of an oil includes consideration of the tendency of the oil to deposit sludge under mild oxidation (72 hr.) and the rate at which this sludge deposition increases as a result of more severe oxidation as the test is continued for 168 and 336 hr.

3. *Apparatus.*—The apparatus shall consist of the following:

(*a*) *Aging* (*Oxidation*) *Bath.*—An oil or wax bath of a suitable type capable of controlling the temperature at 120 ± 0.5 C. with a temperature gradient of less than 1 C.

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[1] These proposed methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials. Published as information, June, 1941.

These proposed methods, while in use, have not been formally adopted. When formally approved, they are intended for incorporation in the Standard Methods of Testing Electrical Insulating Oils (D 117 - 40), 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 90.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 568 (1936), and Vol. 38, Part II, p. 507 (1938), respectively.

in the body of the oil or wax used. Circulation of the oil or wax heating medium such as by a pump or stirrer is recommended.

(*b*) *Drying Tower.*—A drying tube, 10 to 12 in. (25 to 31 cm.) in height, fitted with a ground-glass stopper and side tubes for conditioning of the air supply.[3]

(*c*) *Oil Receptacle and Cover.*—An oil receptacle consisting of a heat-resistant, glass test tube[4] 25 mm. in inside diameter and 150 mm. in length, with a capacity of 50 ml., equipped with a loosely-fitting glass cover having a central hole to admit the air supply tube.

(*d*) *Gooch Crucible.*—A porcelain Gooch crucible, size No. 4, 35-ml. capacity, equipped with a standard quantitative filter paper disk.[5]

(*e*) *Copper Catalyst.*—Commercial copper wire 0.040 in. (1.016 mm.) in diameter.

4. *Petroleum Naphtha.*—Petroleum naphtha, conforming to the requirements prescribed in Section 4 of the Standard Method of Test for Precipitation Number of Lubricating Oils (A.S.T.M. Designation: D 91) of the American Society for Testing Materials,[6] shall be used for dilution of the oxidized oil and precipitation and washing of the sludge.

5. *Preparation of Air Supply.*—The oxidation of the oil shall be obtained by contact with room air conditioned as follows:

(*a*) Dry the air by forcing it through a solid desiccant of high moisture absorbing capacity.[7] Arrange the desiccant in the drying tower to a depth of 8 to 10 in. Change the desiccant frequently (Note).

NOTE.—Desiccants should not be used for a period longer than 1 week.

(*b*) After passing the air through the drying tower, admit it directly to the receptacle containing the oil to be tested. Do not preheat the air.

(*c*) Determine the rate of air supply with a flow meter, and adjust the flow to deliver the conditioned air at a rate of 0.5 ± 0.1 liter per hour to each tube of oil being tested.

(*d*) Rubber connections shall be eliminated as much as possible and when used, the rubber shall be at room temperature. No rubber connections shall be used in contact with the oil or wax of the heating bath or under conditions where the temperature of the rubber will be raised above room temperature. Where it is necessary to use rubber connections, only sulfur-free rubber is permissible.

6. *Preparation of Oil Receptacles.*—Wash each oil receptacle thoroughly first with acetone, then with soap and water, and rinse in chromic acid solution. Wash each receptacle free from chromic acid using tap water, and finally rinse with distilled water. Dry in an air oven at 105 to 110 C. for at least 3 hr., cool to room temperature in a desiccator, and keep the receptacle in the desiccator until ready for use.

7. (*a*) *Preparation of Copper Catalyst.*—Immediately before use, polish the copper wire with No. 00 emery cloth or its equivalent and wipe free from abrasive with a clean dry cloth.

(*b*) Wind a 12-in. (30.5-cm.) length of the polished wire into a helical coil approximately 23 mm. in outside diameter and 2 in. (5.08 cm.) in length. Clean the coil thoroughly with c.p. ethyl ether, air-dry, and insert immediately into the oil receptacle. Handle the clean copper coil only with tongs in order to avoid contamination.

8. (*a*) *Preparation of Gooch Crucible.*—Cover the bottom of the Gooch crucible with the filter paper disk.

(*b*) Prepare a slurry for the formation of the asbestos mat in the Gooch crucible by making a suspension of medium asbestos fiber, Gooch grade, in the proportion of 20 g. of asbestos to 1 liter of distilled water. Pour the asbestos suspension into the Gooch crucible, tamp with a glass rod, and dry by suction. In this manner build up an asbestos mat to a depth of approximately $\frac{1}{16}$ in. (1.59 mm.). Then pour into the crucible, without tamping, additional asbestos suspension to build up the mat to a total thickness of approximately $\frac{1}{8}$ in. (2.17 mm.) after drying by suction.

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[3] A suitable drying tower may be obtained from Eimer and Amend Co., Drying Tube No. 22556.

[4] Pyrex glass is satisfactory for this purpose. A suitable tube conforming to these requirements may be obtained from the Corning Glass Co., Tube No. 9820FZYUV.

[5] A suitable quantitative filter paper disk may be obtained from Carl Schleicher and Schull, designated as white ribbon.

[6] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 72.

[7] Dehydrite is a suitable desiccant for this purpose

(*c*) Place the prepared crucible in an air oven and dry at 105 to 110 C. for 15 to 24 hr., cool in a desiccator, and when needed, weigh to the nearest 0.0001 g.

9. *Conditioning of Sample.*—Filter by gravity the oil to be tested using a previously dried, acid-free filter paper to remove traces of sediment, fibers, and excess water. (Dry the filter paper for 1 hr. at 100 C. before using.) Discard the first 25 ml. of the oil filtrate. Carefully protect the filtrate during the filtration against dust and other contamination.

10. *Procedure.*—(*a*) Prepare three copper catalysts as described in Section 7, insert one each in a clean, dry oil receptacle prepared as described in Section 6, and weigh to the nearest 0.0001 g.

(*b*) Transfer 25 ml. of the conditioned oil sample into each of the three prepared oil receptacles by means of a clean, dry pipette. Protect the oil from contamination by atmospheric moisture and dust during the interval between the placing of the oil in the receptacle and the actual start of the oxidation. Weigh each assembly containing the oil to the nearest 0.0001 g.

(*c*) Adjust the heating bath to maintain a temperature of 120 ± 0.5 C. during the oxidation of the sample.

11. *Oxidation.*—(*a*) Immerse the oil receptacle in the heating bath which has been adjusted to a temperature of 120 ± 0.5 C. Immerse the oil receptacle in the bath to the extent that the surface of the heating bath will be slightly higher than the surface of the test oil in the receptacle. Insert through the holes in the receptacle cover, the clean, dry glass tubes through which the conditioned air is to be led into the oil receptacle. Adjust the tube covers and place the air supply tubes so that they are approximately ½ in. (1.3 cm.) above the oil level in the receptacle and start the air flow through the receptacle. The air shall not be bubbled through the oil. When more than one oil receptacle is fed from a single air supply line, adjust the air flow to each oil receptacle to a rate of 0.5 ± 0.1 liter per hour.

(*b*) Oxidize the three oil samples for periods of 72, 168, and 336 hr., respectively, under the carefully controlled conditions prescribed.

12. *Treatment of Oxidized Oil.*—(*a*) After the required period of oxidation, shut off the air supply and remove the oil receptacle and sample from the oxidizing bath. Cool for 1 hr. to room temperature in a clean dark box or dark room, protecting the oil sample from dust, moisture, or other contamination, and light.

(*b*) At the end of the cooling period, remove the copper catalyst from the oil receptacle and scrape off any sludge adhering to the coil, returning the sludge to the oxidized oil in the receptacle. Wash the copper-wire coil with the petroleum naphtha and transfer the washings to the oxidized oil in the receptacle.

(*c*) Transfer the oxidized oil and naphtha washings to a clean beaker and dilute to 200 ml. at room temperature with the petroleum naphtha. Carefully stir the oil-naphtha mixture until the oil is completely dissolved, cover the beaker, and allow to stand for 1 hr. at a temperature of 25 ± 2 C.

13. *Weighing of Sludge.*—(*a*) Transfer the oil-naphtha solution to the prepared, dried, and weighed Gooch crucible. Also wash any sludge adhering to the oil receptacle free from oil with naphtha, dry and scrape free, and transfer to the crucible by means of a suitable brush. Determine the weight of any sludge that cannot be removed from the oil receptacle from the difference in weight of the sludged receptacle (cleaned to remove bath oil or wax, and dirt from the outside) and the weight of the receptacle after cleaning free from sludge as described in Section 6. Add the weight of the sludge remaining in the receptacle to the weight of the sludge in the Gooch crucible.

(*b*) Wash the sludge in the Gooch crucible thoroughly with naphtha to remove all traces of adhering oil. Normally three washes of 25-ml. portions each are sufficient. A drop of the final naphtha washing should show substantially no oil stain on the filter paper.

(*c*) Dry the sludge in the Gooch crucible by suction and place in an air oven at 105 to 110 C. for 1 hr., cool to room temperature in a desiccator, and weigh to the nearest 0.0001 g.

14. *Calculation and Report.*—(*a*) Calculate the percentage of total sludge in the oil as follows:

$$\text{Sludge value} = \frac{\text{wt. of total sludge}}{\text{wt. of oil}} \times 100$$

(*b*) Report the sludge value for each of the test periods of 72, 168, and 336 hr.

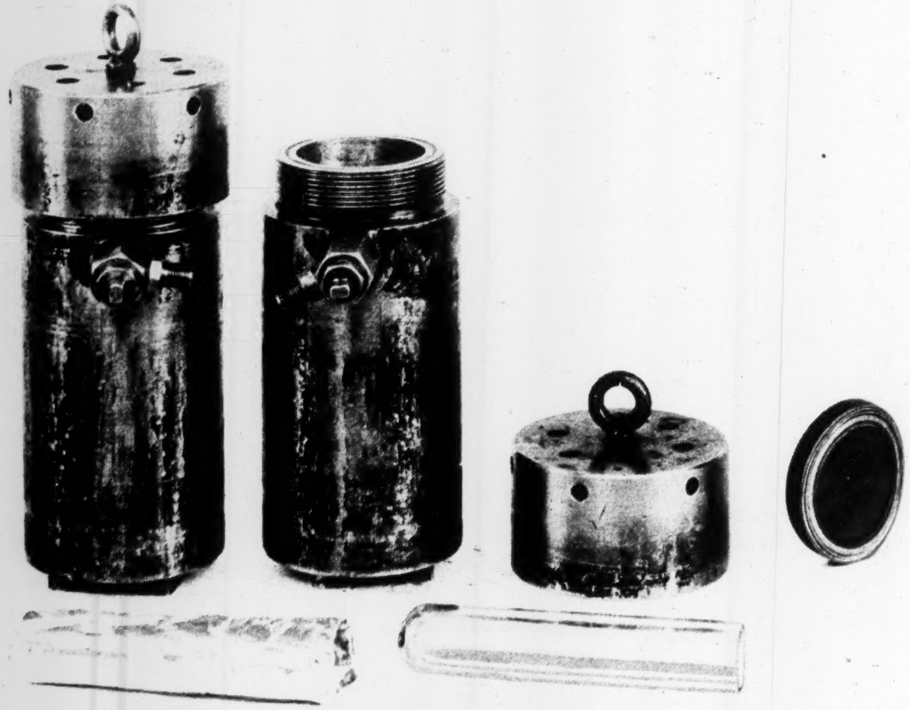

FIG. 1.—Bomb and Apparatus for Determining Bomb Sludge Value of Mineral Transformer Oils.

Method B: High-Pressure Oxidation Test

15. *Nature of Test.*—The high-pressure oxidation test determines the "Bomb Sludge Value", which is the percentage by weight of sludge formed after 24-hr. oxidation at 140 C. under 250 psi. of oxygen pressure.

16. *Apparatus.*—The apparatus shall consist of the following:

(*a*) *Aging* (*Oxidation*) *Bath.*—An oil or wax bath of a suitable type capable of controlling the temperature at 140 ± 0.5 C. with a temperature gradient of less than 1 C. in the body of the oil or wax used. Circulation of the oil or wax heating medium such as by a pump or stirrer is recommended. The heat source shall have sufficient capacity to return the bath to the required temperature within 30 min. after immersion of the cold bomb.

(*b*) *Bomb.*—A pressure bomb of the type shown in Fig. 1, conforming to the dimensions prescribed in Fig. 2. The bomb shall be made of stainless steel, capable of withstanding with safety an internal pressure of 10,000 psi., and provided with a lead gasket for sealing.

(*c*) *Oil Receptacle.*—An oil receptacle consisting of a heat-resisting, centrifuge-type tube,[8] $1\frac{1}{8}$ in. (28.6 mm.) in inside diameter, and 6 in. (152.4 mm.) in length, with a capacity of 100 ml.

(*d*) *Gooch Crucible.*—A porcelain Gooch crucible, size No. 4, 35-ml. capacity, equipped with a standard quantitative filter paper disk.[5]

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[8] Pyrex glass is satisfactory for this purpose. A suitable tube conforming to these requirements may be obtained from the Corning Glass Co., Tube No. 8240FIUGL.

17. *Oxidizing Gas.*—For oxidizing the oil, pure oxygen gas under a gage pressure of 250 psi. at the oxidation temperature of 140 C. shall be used.

18. *Petroleum Naphtha.*—The petroleum naphtha prescribed in Section 4 shall be used for the dilution of the oxidized oil and the precipitation and washing of the sludge.

19. *Preparation of Bomb.*—Clean and dry the high-pressure bomb before using. To insure cleanliness, rinse the bomb with the petroleum naphtha, drain in air, and dry in an air oven at 105 to 110 C. for 3 hr. Cool the bomb to a temperature of 25 ± 2 C. just before using.

20. *Preparation of Oil Receptacle.*—Prepare the oil receptacle in accordance with the procedure described in Section 6.

21. *Preparation of Gooch Crucible.*—Prepare the Gooch crucible in accordance with the procedure described in Section 8.

22. *Conditioning of Sample.*—Filter by gravity the oil to be tested using a previously dried, acid-free filter paper to remove traces of sediment, fibers, and excess water. Dry the filter paper for 1 hr. at 100 C. before using. Discard the first 25 ml. of the oil filtrate. Carefully protect the filtrate during the filtration against dust and other contamination.

23. *Precautions.*—(*a*) No oxygen gas pressure in excess of 250 psi. shall be applied to the bomb containing the oil sample. Under no conditions shall oxygen gas be admitted to the bomb except at room temperature. Any bomb which develops a gas leak during the oxidation procedure shall immediately be removed as described in the note in Section 25 (*b*).

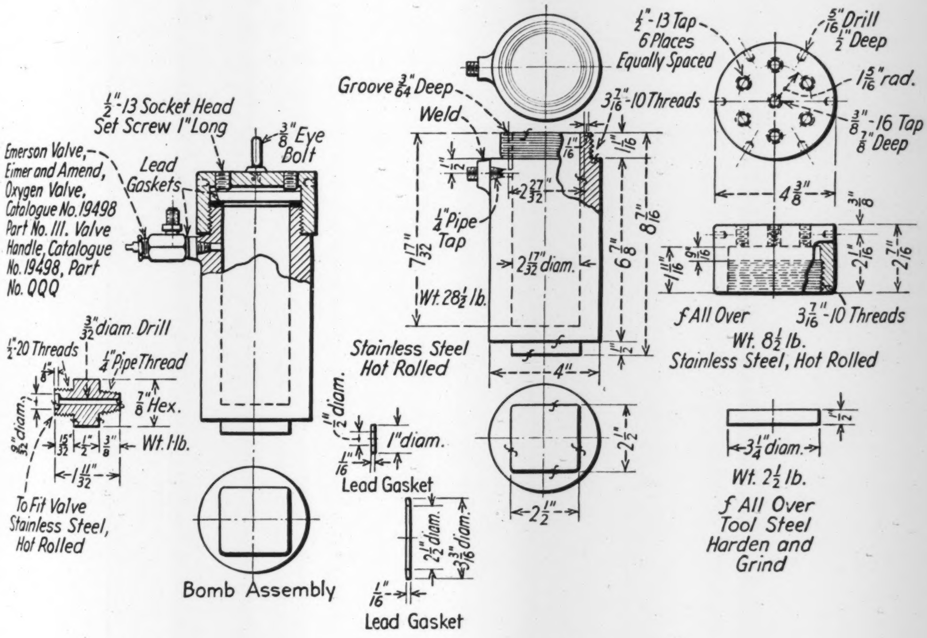
1/2"-13 Socket Head Set Screw 1" Long
3/8" Eye Bolt
Emerson Valve, Eimer and Amend, Oxygen Valve, Catalogue No. 19498 Part No. III. Valve Handle, Catalogue No. 19498, Part No. QQQ
Lead Gaskets
3/32" diam. Drill
1/2"-20 Threads
1/4" Pipe Thread
1/8"
7/8" Hex.
9/32" diam.
15/32"
1/2"
3/8"
Wt. 1 lb.
1 11/32"
To Fit Valve Stainless Steel, Hot Rolled
Bomb Assembly
Groove 3/64" Deep
Weld
1/2"
1/4" Pipe Tap
7 17/32"
Wt. 28 1/2 lb.
2 27/32"
2 17/32" diam.
3 7/16"-10 Threads
1/16"
1 1/16"
6 7/8"
8 7/16"
1/2"
4"
Stainless Steel Hot Rolled
1/2" diam.
1" diam.
1/16"
Lead Gasket
2 1/2" diam.
3 3/16" diam.
1/16"
Lead Gasket
2 1/2"
2 1/2"
1/2"-13 Tap 6 Places Equally Spaced
5/16" Drill 1/2" Deep
1 5/16" rad.
3/8"-16 Tap 7/8" Deep
4 3/8"
3/8"
9/16
1 11/16"
2 1/16"
2 7/16"
f All Over
3 7/16"-10 Threads
Wt. 8 1/2 lb.
Stainless Steel, Hot Rolled
1/2"
3 1/4" diam.
Wt. 2 1/2 lb.
f All Over Tool Steel Harden and Grind

FIG. 2.—Detailed Dimensions of Steel Bomb.

(*b*) Under no condition shall a bomb containing oil under oxygen gas pressure be handled carelessly. It must not be dropped or struck with a metal object. Tapping, if necessary for any purpose, shall be done with a wooden or fiber hammer.

24. *Procedure.*—(*a*) Weigh to the nearest 0.0001 g. the clean and dry oil receptacle, prepared as described in Section 6.

(*b*) Transfer 50 ml. of the conditioned oil sample into the prepared oil receptacle by means of a clean, dry pipette. Protect the oil from contamination by atmospheric moisture and dust during the interval between the placing of the oil in the receptacle and its closure in the bomb. Weigh the prepared receptacle containing oil to the nearest 0.0001 g.

(*c*) Place the oil receptacle containing the weighed amount of conditioned oil in the bomb in a vertical position using a suitable wire rack or holder. Set the bomb cover in position and tighten it using a lead gasket. When the bomb has been properly sealed, flush once by filling with oxygen to a pressure of 25 psi. and slowly releasing the pressure to atmospheric pressure.

(*d*) After flushing, admit the oxygen gas at room temperature to the assembled bomb containing the conditioned oil. Apply the gas pressure so as to produce a gage pressure of 180 psi. at 25 C. This will give the required gas pressure of 250 psi. at the oxidation temperature of 140 C. The oxygen gas must not be applied to the assembled bomb at any temperature above room temperature.

(*e*) Adjust the heating bath to maintain a temperature of 140 ± 0.5 C. during the oxidation of the sample.

25. *Oxidation.*—(*a*) When the pressure of 180 psi. of oxygen gas in the bomb has been obtained, suspend the bomb immediately in the wax or oil bath which has been adjusted to a temperature of 140 ± 0.5 C. The temperature of the bath will drop when the bomb is immersed. Regulate the heat supply so as to return the temperature to 140 C. within 30 min. after immersion. Totally immerse the charged bomb in the heating bath.

(*b*) Record the exact time at which the bomb is immersed. Continue the oxidation period for exactly 24 hr., maintaining the temperature of the heating bath at 140 ± 0.5 C. (Note).

NOTE.—After the charged bomb has been immersed in the heating bath or its temperature otherwise raised above room temperature, the gas pressure shall not be adjusted or changed in any manner except for unloading the bomb at the end of the oxidation period. No change in gas pressure shall be made while the bomb is immersed in the heating bath. If the gas pressure falls because of leaks, the bomb shall be removed from the heating bath, the gas pressure reduced to atmospheric pressure, the bomb unloaded, and the oil sample discarded. When once the gas pressure has been released, partially or totally, there shall be no reapplication of the gas pressure.

26. *Treatment of Oxidized Sample.*—(*a*) Exactly 24 hr. after the bomb has been placed in the heating bath, remove it from the bath and slowly release the pressure to atmospheric pressure. Remove the bomb cover and cool the oil receptacle for 1 hr. to room temperature in a clean dark box or dark room, protecting the oil sample from dust, moisture, or other contamination, and light.

(*b*) Transfer the oxidized oil to a clean beaker and dilute to 200 ml. at room temperature using the petroleum naphtha. Carefully stir the oil-naphtha mixture until the oil is completely dissolved, cover the beaker, and allow to stand at a temperature of 25 ± 2 C. for 1 hr.

27. *Weighing the Sludge.*—Weigh the sludge obtained in accordance with the procedure described in Section 13.

28. *Calculation and Report.*—(*a*) Calculate the bomb sludge value as follows:

$$\text{Bomb sludge value} = \frac{\text{wt. of total sludge}}{\text{wt. of oil}} \times 100$$

(*b*) Report the bomb sludge value as determined.

REPORT OF SECTIONAL COMMITTEE
ON
ELECTRICAL INSULATING MATERIALS

ASA Project: C59

Sectional Committee C59 on Electrical Insulating Materials held one meeting during the year: in Washington, D. C., on March 3, 1941.

Since the organization of Sectional Committee C59 in 1934, the following A.S.T.M. standards have been submitted to the American Standards Association on the recommendation of the sectional committee and have been approved as American standard:

Standard Methods of:
- Testing Molded Materials Used for Electrical Insulation (A.S.T.M. D 48 - 39; ASA C59.1-1940),
- Testing Electrical Insulating Oils (D 117 - 40; C59.2-1941),
- Test for Insulation Resistance of Electrical Insulating Materials (D 257 - 38; C59.3-1939),
- Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 - 38; C59.10-1941),

Standard Specifications for:
- Rubber Matting for Use Around Electrical Apparatus or Circuits not Exceeding 3000 Volts to Ground (D 178 - 24; C59.4-1935), and
- Rubber Insulating Tape (D 119 - 38; C59.6-1939).

The A.S.T.M. Standard Methods of Test for Impact Resistance of Electrical Insulating Materials (D 256 - 38) have now been approved by letter ballot in Sectional Committee C59 and will shortly be submitted to ASA for approval as American standard.

The following A.S.T.M. standards are now being reviewed by special committees looking toward their submittal to the ASA for approval as American standard:

Methods of Testing:
- Laminated Tubes Used in Electrical Insulation (D 348 - 39)
- Laminated Round Rods Used in Electrical Insulation (D 349 - 39),
- Shellac Used for Electrical Insulation (D 411 - 40), and

Standard Specifications for:
- Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground (D 120 - 40).

The following standards submitted by the National Electrical Manufacturers Assn. are likewise being reviewed by special committees looking toward their submittal to the ASA for approval as American standards:

- Manufactured Electrical Mica Standards (N.E.M.A. 39-55),
- Laminated Phenolic Products Standards (N.E.M.A. 39-57), and
- Recommended Practice for Machining and Punching of Laminated Phenolic Plates (N.E.M.A. 39-58).

Although it is becoming more and more difficult to get answers from the central office of the International Electrotechnical Commission in London, Sectional Committee C59 is still following the activities of their Committee No. 10 on Insulating Oils through the technical adviser to the U. S. National Committee, E. A. Snyder. This committee is trying to develop a universal sludging test. A set of samples of oil taken from five installed transformers in Holland has been received and the United States committee has in turn sent samples of the oils on which it has been working to Dr. Bruckman in Holland. Results of the tests on these oils are not yet available.

Mr. W. H. Gardner, technical adviser on I. E. C. Committee No. 15 on Shellac, receives brief monthly reports indicating that work is still being carried on. A large part of the work is of a defense nature, however, and details are not available except through our own Government channels.

A subcommittee under the chairmanship of Mr. G. E. Landt has completed the preparation of a preliminary list containing the titles and serial designations of the available standards in the field of electrical insulating materials. This list contains only those standards that have been issued by nationally recognized organizations. This effort represents the first step in compiling a library of available standards for electrical insulating materials to be deposited in the office of the secretary, and to provide a cross-section of existing standards in this field. The list is now being carefully reviewed to assure its completeness. With the list available, Committee C59 will be in a position to initiate work in any part of the field that appears to be not adequately covered. Also steps can be taken looking toward the submittal of any appropriate standards to the ASA for approval as American standard.

Respectfully submitted on behalf of the sectional committee,

H. L. CURTIS,
Chairman.

R. E. HESS,
Secretary.

REPORT OF COMMITTEE D-10
ON
SHIPPING CONTAINERS

During the past year there have been no meetings of Committee D-10 on Shipping Containers. The chairman and the secretary of the committee have met and discussed the possibilities of increasing the usefulness of this committee. Several new members have been added, and it is the plan of the committee to set up acceptable and standard procedures for making tests on shipping containers. One purpose of expanding the committee was to add more consumer members who could advise the committee of their needs in setting up the standard test procedures.

The first general division of tests would be those on the whole container. Under this division subcommittees would be formed to report to the entire committee on such tests as:

7-ft. Revolving Drum,
14 ft. Revolving Drum,
Incline Impact Tester,
Compression Test,
Drop Test,
Swing Test, and
Special Impact Tests.

The standards set up for these various tests should give a measure of the following:

1. The retention-of-contents quality of a complete container,
2. The protection-of-contents quality of the complete container,
3. The protection-to-crushing quality of the complete container, and
4. The effectiveness of various constructional features.

The second division of tests would be on materials and accessories used in the construction and packing of shipping containers, omitting paper and paper products now covered by Committee D-6 on Paper and Paper Products. These would include both static and dynamic tests.

It has not been possible as yet for this committee to set up its various subcommittees, but plans are being made to do this in the near future. There is every reason to believe that, in view of the present situation in world affairs and national defense, considerable effort expended in setting up uniform measuring procedures for the qualities of shipping containers will yield favorable returns to both industry and Government.

This report has been submitted to letter ballot of the committee which consists of 17 members; 12 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

EDWARD DAHILL,
Chairman.

E. R. STIVERS,
Secretary.

REPORT OF COMMITTEE D-11

ON

RUBBER PRODUCTS

Committee D-11 on Rubber Products has met only once during the year, but its work has been carried on in the numerous subcommittees and by correspondence and has been substantially advanced. The committee meeting was held in Washington, D. C., on March 6, 1941, in connection with the spring group meetings of A.S.T.M. committees.

Subsequent to the 1940 annual meeting, Committee D-11 presented to the Society through Committee E-10 on Standards the following recommendations:

Revisions of Tentative Methods of:

Test for Compression Set of Vulcanized Rubber (D 395 - 40 T),

Tension Testing of Vulcanized Rubber (D 412 - 39 T),

Testing Rubber Insulated Wire and Cable (D 470 - 39 T),

Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 - 40 T),

Revision of Tentative Specifications for:

Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 - 40 T),

Tentative Revisions of Standard Methods of:

Sample Preparation for Physical Testing of Rubber Products (D 15 - 39), and

Chemical Analysis of Rubber Products (D 297 - 39), in the form of new Tentative Methods of Chemical Analysis of Rubber Products (D 297 - 40 T).

These recommendations were accepted[1] by Committee E-10 on August 28, 1940, and appear in the 1940 Supplement to the Book of A.S.T.M. Standards, Part III. At the same time, Committee D-11 recommended to Committee E-10 three additional new tentative standards, as follows:

Methods of:

Test for Resistance to Accelerated Light Aging of Rubber Compounds,

Calibrating a Light Source Used for Accelerating the Deterioration of Rubber, and

Test for Tear Resistance of Vulcanized Rubber.

Because of the number of negative votes, these methods were not accepted as tentative but were approved for publication as information.[2]

On September 26, 1940, Committee E-10 accepted for publication a new Tentative Method of Test for Physical State of Cure of Vulcanized Rubber (T-50 Test), and approved the withdrawal of the Standard Methods of Chemical Analysis of Rubber Products (D 297 - 39). The new tentative method appears in the 1940 Supplement to the Book of A.S.T.M. Standards, Part III, bearing the designation D 599 - 40 T.

The T-50 test method for physical state of cure fills a need for direct determination of a property of great importance in the application of vulcanized rubber. Prior to the development of this method in the laboratories of the United States Rubber Co. several years ago, no method was available for comparing the physical states of cure of a rubber compound after vulcanization by various procedures except indirectly from tension tests on specimens cured over the range of times and temperatures. The test has been fully described in the literature[3] and has attained widespread use. These tentative methods, however, are the first presentation in standardized form.

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[1] In submitting these recommendations to Committee E-10 on Standards, Committee D-11 reported the following results of the letter ballot vote of a total of 70 ballots returned from a committee membership of 112: D 395, affirmative 46, negative 0, ballots marked "not voting" 24; D 412, affirmative 60, negative 0, ballots marked "not voting" 10; D 470, affirmative 40, negative 0, ballots marked "not voting" 30; D 471, affirmative 45, negative 0, ballots marked "not voting" 25; D 469, affirmative 38, negative 1, ballots marked "not voting" 31; D 15, affirmative 59, negative 0, ballots marked "not voting" 11; D 297, affirmative 56, negative 0, ballots marked "not voting" 14.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 381, 385, 389 (1940).

The new tentative methods of chemical analysis represent an extensive revision of the former methods which they replace. They are a marked improvement and have been in the process of development over a period of several years. A large amount of work has been done as covered from time to time in the committee reports, and additional work is being continued. It was considered, however, that these analytical procedures were sufficiently advanced to be published as tentative methods.

The proposed revisions in tentative standards represent changes which have been found by experience to be needed for improvement and to bring them in line with best practice. The changes in the methods for compression set (D 395 - 40 T) include refinements in Method B, the test under constant deflection, and a new method of calculation whereby the set as determined will be reported in terms of percentage of original deflection rather than of original thickness of specimen. The change in the methods of tension testing (D 412 - 40 T) deals with the discarding of erratic results and changes the checking requirement from 5 per cent to 10 per cent of the highest value obtained. The changes in the method of testing insulated wire and cable (D 470 - 40 T) refer particularly to the test for ozone resistance. They provide for better temperature regulation during the test and for improvement in the determination of ozone concentration. The revisions in the methods of test for changes in liquids (D 471 - 40 T) provide for the use of dumbbell shaped specimens for immersion when tension tests are to be made and change the method of calculation in the case of such tests as well as reducing the recovery period prior to test after removal from the liquid. These revisions were the result of extensive cooperative tests conducted by five laboratories. The revision of the specification for insulated wire and cable: heat resisting compound (D 469 - 40 T) removes the requirement for the 120 hr. air oven test at 121 C. and brings Table II in line with jurisdiction of the National Electrical Code. The tentative revision of the methods of sample preparation (D 15 - 39) affects the section dealing with buffing of specimens and brings the A.S.T.M. method into agreement with the Federal Specification. Additional approved equipment is also listed.

The revised Standard Specifications for Cotton Rubber-Lined Fire Hose for Public and Private Fire Department Use (D 296 - 38) have now been reapproved as American standard (ASA L3.1-1941) by the American Standards Association.

Committee D-11 has submitted the Standard Specifications for Rubber Gloves for Electrical Workers on Apparatus or Circuits not Exceeding 3000 Volts to Ground (D 120 - 40) to Sectional Committee C59 on Electrical Insulating Materials for review looking toward their submittal to the American Standards Association for approval as American standard.

At its March meeting, Committee D-11 voted to recommend the following to the ASA for approval as American standards, after adoption this year by the A.S.T.M. of revisions in the last four methods in the list:

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[3] W. A. Gibbons, R. H. Gerke, and H. C. Tingey, "The T-50 Test for State of Cure," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 5, No. 4, July 15, 1933, p. 279.

Tentative Methods of:

Chemical Analysis of Rubber Products (D 297 – 40 T),
Testing of Rubber Hose (D 380 – 40 T),

Standard Methods of:

Sample Preparation for Physical Testing of Rubber Products (D 15),
Tension Testing of Vulcanized Rubber (D 412),
Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572), and
Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573).

I. NEW TENTATIVE STANDARDS

Tentative Methods of Testing Automotive Air Brake and Vacuum Brake Hose.[4]—These methods have been developed by Technical Committee A on Automotive Rubber in response to urgent requests of automotive engineers and state highway departments for suitable standards for the control of the brake hose used on licensed vehicles on the highways. The status of air brake hose and vacuum brake hose is similar to that of hydraulic brake hose for which suitable methods were approved last year. The use of inferior or defective hose on vehicles equipped with these braking systems is especially serious from a safety viewpoint because of their widespread application to automotive vehicles and to heavy trucks in particular. As with hydraulic brake hose, Technical Committee A has prepared standard purchase specifications which will be offered to the Society of Automotive Engineers for inclusion among its standards, and the methods of test for these materials are herewith submitted to the A.S.T.M. with the recommendation that they be accepted for publication as tentative.[4]

Tentative Method of Test for Tear Resistance of Vulcanized Rubber.[5]—Since this method was submitted to letter ballot last year, the section dealing with its scope has been completely revised and rewritten so as to define more accurately its limitations. In addition, a change has been made in the number of nicks in the test specimen which is reduced from five to one. In establishing this change, cooperative tests have been carried out in Subcommittee XIV and the data obtained show better reproducibility of the test when the specimens have a single nick. In addition, the relation between the two styles of specimens permitted has been established and is included in the method. A suitable standardized method for testing tear resistance of rubber is much needed and has been specifically requested by Technical Committee A on Automotive Rubber. While any relation between the results of the test here presented and the actual service value of the rubber being tested is not well established, the method is widely used in research and in comparative test work and may be considered as the best method at present available. The committee recommends that the method be accepted for publication as tentative.[5]

Tentative Methods of Test for Compression Fatigue of Vulcanized Rubber.[6]—These methods have been prepared by Subcommittee XVIII on Dynamic Fatigue tests and were discussed under the activities of this subcommittee in the annual report of Committee D-11 on Rubber Products for 1939. Since then the subcommittee has done additional work and has compared these methods with each other and with other tests on the same rubber stocks. The results in general were found to be consistent. Since the methods are fairly new, their reproducibility in various laboratories has not as yet been fully investigated and they should not be used in purchase specifications. However, they have been found so advantageous for research and development purposes in comparing properties which can not be determined otherwise that the committee believes they should be published as tentative at this time pending completion of further work.

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[4] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 504.

[5] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 493.

[6] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 496.

II. REVISION OF TENTATIVE STANDARD

Tentative Methods of Testing Sponge Rubber Products (D 552 - 39 T).[7]—The committee is proposing the following revision in these tentative methods and recommends that as revised they be continued as tentative:

Section 2(a).—Under the paragraph on aging test, add a reference to the "Standard Method for Air Pressure Heat Test of Vulcanized Rubber (A.S.T.M. Designation: D 454)."

Section 6 (d).—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

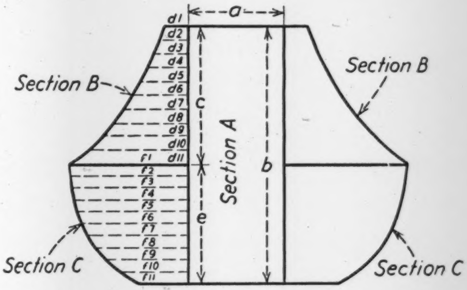
> The volume of irregularly shaped products or specimens which cannot be measured by ordinary [methods] *means* shall be obtained by *one of* the following [displacement method] *alternate methods:*

Redesignate the remainder of this paragraph as "*(1)* Displacement Method," and add the following alternate method:

> *(2) Calculation Method.*—An alternative method of obtaining the volume of irregularly shaped articles is to obtain an average dimension of an irregularly shaped section by dividing the section into ten smaller segments of equal width, making eleven measurements, and dividing by eleven to obtain the average dimension. The volume is then calculated using the average dimensions obtained. This method is illustrated in Fig. 2 (accompanying Fig. 1).

Section 13.—Change the first sentence to read as follows:

> Either the oxygen-pressure-chamber aging test, the air-oven aging test, or the air pressure heat test as described in the Standard Methods of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (A.S.T.M. Designation: D 572), by the Oven Method (A.S.T.M. Designation: D 573), or by the Air Pressure Heat Test Method (A.S.T.M. Designation: D 454), respectively, may be used for sponge rubber, as specified, except that in the air pressure heat test an air pressure of 60 ± 2 psi. is usually used in place of the 80 ± 2 psi. prescribed in Methods D 454.



Area of section A = $a \times b$

Area of section B = $c \times \left(\frac{d1 + d2 \cdots d11}{11}\right)$

Area of section C = $e \times \left(\frac{f1 + f2 \cdots f11}{11}\right)$

Total area = area A + 2 area B + 2 area C

Volume = total area × thickness

Fig. 1.—Calculation Method for Obtaining Volume of Irregularly Shaped Articles.

Section 15.—Change the heading preceding Section 15 from "Compression Tests" to read "Compression Deflection Tests."

Section 16 (b).—Add the following as the next to the last sentence in this paragraph:

> The reading shall be taken 1 min. after the load has been applied.

Section 24 (a).—Change the last sentence from its present form: namely,

> The specimen position shall be such that the indentation will be made at the location agreed upon by the manufacturer and the purchaser,

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[7] 1939 Book of A.S.T.M. Standards, Part III, p. 931.

but in the absence of such agreement, the following locations shall be used:

Type of Surface	Location of Indentor Foot
Crown (convex) surface	highest point
Saddle (concave) surface	lowest point
Combination crown and saddle	highest point
Flat products	dead center
Double contours	test contour on larger section
Head roll	highest point

to read as follows:

> The specimen position shall be such that the indentation will be made at the center of all articles.

III. REVISIONS OF STANDARDS

Standard Methods of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 – 40),[8] *Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 – 40),*[8] *and Air Pressure Heat Test of Vulcanized Rubber (D 454 – 40).*[8]—The committee recommends for immediate adoption in each of these methods a revision as indicated below, and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that this modification may be referred to letter ballot of the Society. This revision will change the provision for averaging tension results including the highest and any which check within 10 per cent to the average of the highest and any which check within 15 per cent. The reason for recommending this change is that results of tension tests on rubber specimens, which have been submitted to the drastic aging treatment, are somewhat more variable than similar tests on unaged specimens. In practice, therefore, it has been found that adherence to the 10 per cent limit has resulted in the discarding of significant data. The committee believes that the 15 per cent value is a better tolerance.

Section 9.—Change the second sentence to read as follows by the addition of the italicized figure and the omission of that in brackets:

> In determining the physical properties after aging, the final values shall be the average of results from at least two specimens including that one giving the highest value for ultimate tensile strength and any which check that value within [10] *15* per cent.

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the tentative standards listed below be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Methods of Testing Flat Rubber Belting (D 378 – 40 T).[9]—These methods have stood in the tentative status for three years with only minor revisions. No criticism has been received during the past year, and it is believed that the methods are now satisfactory for adoption as standard.

Tentative Method of Test for Indentation of Rubber by Means of the Pusey and Jones Plastometer (D 531 – 39 T).[10]—This method has stood in the tentative status, without revision since it was issued two years ago. No criticism has been received. The method is substantially the same as given in the Federal Specification ZZ-R-601a covering general methods of testing rubber goods.

Tentative Methods of Testing Rubber Insulated Wire and Cable (D 470 – 40 T).[11]—These methods were issued in 1937 and have since undergone a number of important revisions. They are now widely used in the wire and cable industry and, in the opinion of the committee, are ready for adoption as standard since no criticism has been received during the past year.

Tentative Methods of Tension Testing

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[8] 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 102, 107, 111.

[9] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 365.

[10] 1939 Book of A.S.T.M. Standards, Part III, p. 855.

[11] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 410.

of Vulcanized Rubber (*D 412 – 40 T*),[12] revised as follows:

Figure 3.—Add an illustration of a new die designated as "Die D" as shown in the accompanying Fig. 2.

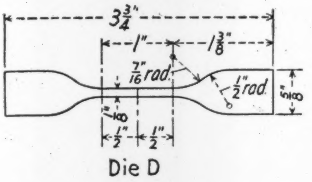

Fig. 2.—Die D Specimen.

Section 8.—In the first sentence change the phrase "specimens shall conform in shape to die A, B, or C" to read "specimens shall conform in shape to die A, B, C, or D."

Change Paragraph (*b*) from its present form to read as follows:

(*b*) Die B or die C shall be used in all cases where the dimensions of the sample and the capacity of the testing machine permit except when otherwise specified in detailed specifications or test methods covering a particular product which shall take precedence. Die B shall be used only in preparing specimens from compounds having high filler content. When the dimensions or form of a sample are such as to preclude the preparation of adequate specimens using die B or die C, die D shall be used. In all cases in which the sample and the testing machine limitations permit a choice in die shapes, the die giving the higher test values shall be used as such higher results will be nearer the true value of the material.

Add the following new sentence at the end of Paragraph (*e*):

In the case of specimens prepared with die D, the measurement of thickness shall be taken with the center of the micrometer foot coinciding with the longitudinal center line of the specimen so that there is an equal overlap of the foot on either side of the specimen.

Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (*D 27 – 40 T*),[13,14] revised as follows:

Table V.—Change the minimum conductor sizes from "No. 14 to No. 9" for the voltage rating of 0 to 600 v. to read "No. 16 to No. 9,[a]" including the addition of the following footnote:

[a] Sizes Nos. 16 and 18 are not recognized by the National Electrical Code for 600 v. service.

Change Note 4 to read as follows by the addition of the italicized words:

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits *except where one side of a delta system is permanently grounded, in which case, multiply the circuit voltage* (*phase to phase*) *by 1.73 and use the resulting voltage to select the corresponding insulation thickness.*

Tentative Specifications for Insulated Wire and Cable: Performance Rubber Compound (*D 353 – 40 T*),[15] revised as follows:

Table II.—Make the same changes in this table and in Note 4 as are recommended above in Table V of Specifications D 27 – 40 T.

Tentative Specifications for Insulated Wire and Cable: Heat-Resisting Rubber Compound (*D 469 – 40 T*),[16] revised as follows:

Table II.—Make the same changes in this table and in Note 4 as are recommended above in Table V of Specifications D 27 – 40 T.

Tentative Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables (*D 532 – 39 T*),[17] revised as follows:[18]

Section 8 (*b*).—Omit the word "minimum" before the word "average."

V. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

The committee recommends that the tentative revision of the Standard Methods of Sample Preparation for

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[12] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 320.

[13] See Editorial Note, p. 431.

[14] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 374.

[15] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 404.

[16] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 390.

[17] 1939 Book of A.S.T.M. Standards, Part III, p. 914.

[18] The recommendation for adoption as standard of these tentative specifications failed of approval at the annual meeting of the Society, see Summary of Proceedings, p. 15. The proposed modification in Section 8 (*b*) has been made as an editorial change in the specifications.

Physical Testing of Rubber Products (D 15 – 39),[19] published last year, dealing with buffing of specimens, be approved for reference to letter ballot of the Society for adoption as standard since no criticisms have been received.

VI. Tentative Standards Continued without Revision

The committee recommends that all tentative standards under its jurisdiction not mentioned in this report be continued as tentative without revision. The Tentative Method of Test for Resistance to Light Checking and Cracking of Rubber Compounds (D 518 – 38 T) has had a tentative status for three years without revision. The committee believes that some changes are desirable and an effort will be made to design a practical method for stretching the specimens more uniformly and for mounting them more conveniently. The Tentative Methods of Test for Viscosity and Total Solids Content of Rubber Cements (D 553 – 39 T) have been in the tentative status for two years. These methods are to be continued tentative as further attention is being given to other procedures for determining viscosity.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 117 members; 75 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative Standards			
Testing Automotive Air Brake and Vacuum Brake Hose	32	0	43
Test for Tear Resistance of Vulcanized Rubber	51	1	23
Test for Compression Fatigue of Vulcanized Rubber	40	0	35
II. Revision of Tentative Standard			
Testing Sponge Rubber Products (D 552 – 39 T)	38	1	36
III. Revisions of Standards			
Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 – 40), immediate adoption	64	0	11
Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573 – 40), immediate adoption	64	0	11
Air Pressure Heat Test of Vulcanized Rubber (D 454 – 40), immediate adoption	64	0	11
IV. Adoption of Tentative Standards as Standard			
Testing Flat Rubber Belting (D 378 – 40 T)	35	0	40
Tension Testing of Vulcanized Rubber (D 412 – 40 T), as revised	62	0	13
Test for Indentation of Rubber by Means of the Pusey and Jones Plastometer (D 531 – 39 T)	45	0	30
Testing Rubber Insulated Wire and Cable (D 470 – 40 T)	42	1	32
Spec. for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 40 T), as revised	38	1	36
Spec. for Insulated Wire and Cable: Performance Rubber Compound (D 353 – 40 T), as revised	38	1	36
Spec. for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 40 T), as revised	36	2	37
Spec. for Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 – 39 T), as revised[a]	33	2	40
V. Adoption as Standard of Tentative Revision of Standard			
Sample Preparation for Physical Testing of Rubber Products (D 15 – 39)	63	0	12

[a] The recommendation for adoption as standard of these tentative specifications failed of approval at the annual meeting of the Society, see Summary of Proceedings, p. 15.

Activities of Subcommittees

Interest in the work of Committee D-11 and the activities of the various subcommittees has been well maintained during the year. A new subcommittee to develop standard methods of test for proofed fabrics was authorized and A. F. Schildhauer has been appointed chairman. Although Committee D-13 on Textile Materials has already developed Specifications and Methods of Test for Cotton Goods Used for Rubber and Pyroxylin Coating (A.S.T.M. Designation: D 334), standard test methods for various finished products made from fabrics which are coated with rubber and rubber-like materials are urgently needed and are considered to be within the scope of Committee D-11. Numerous tests are now included in the specifications for these materials, but there is very little standardization and in some cases the tests leave much to be desired.

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[19] 1939 Book of A.S.T.M. Standards, Part III, p. 331.

Technical Committee A on Automotive Rubber (L. A. Danse, chairman).—This technical committee has actively advanced its work along lines indicated in the 1940 report. A comprehensive review of its accomplishments has been published in the ASTM BULLETIN.[20] The committee has held six meetings in Detroit, Mich., during the year. The section on motor mountings completed an extensive cooperative test investigation on compression set testing in which samples from seven sources in five hardness grades were tested in ten cooperating laboratories. Over 1400 individual determinations were made and the data obtained supports the following conclusions:

> 1. Satisfactory duplication of results between laboratories is accomplished using either the A or B method of the Tentative Methods of Test for Compression Set of Vulcanized Rubber (D 395 - 40 T).[21]
>
> 2. Results obtained by Method B lend themselves to more useful interpretation when considering several hardnesses of compounds and should be used in the standard classification of motor mounting stocks.
>
> 3. Factors have been developed whereby Method A values can be converted to corresponding Method B values.

In connection with this test program, considerable difference of opinion developed concerning the values for compression set originally selected for use in the proposed standard S.A.E. classification, and the fact was brought out that some of these values are very difficult to meet, as well as not being necessary in the manufacture of certain mountings which have proven satisfactory in service. Also, some of the compounds which have the lowest set values will not adhere directly to metal surfaces but require an intermediate layer referred to in the industry as tie gum. Such compounds are suitable for motor mountings but are more costly to manufacture and possibly do not justify the extra cost in better service value. It was finally agreed to recommend two classifications over the hardness range, one classification permitting higher set values but including requirements as to tensile strength and elongation of the rubber, the other classification remaining substantially as prepared last year. These classifications were approved for recommendation as S.A.E. standard. The work on the production type of compression testers and on portable hardness testing instruments has been continued. Efforts were made to set up a standard means of calibrating a Shore durometer, but these have not yet been successful because no satisfactory standard is available to calibrate against. The section is now endeavoring to obtain such a standard.

The Section on Bumpers has continued its work on load deflection, impact tests, and heat failure of bumper stocks. A large amount of work has been done, but the section is not yet ready to recommend either classifications or test procedures.

Section III, on Hydraulic Brake Hose, which last year completed specifications and methods of test for hydraulic brake hose, has this year presented the proposed Tentative Methods for Testing Automotive Air Brake and Vacuum Brake Hose,[4] together with specifications which are being recommended as S.A.E. standard. Work is being continued on the development of test methods applicable to hose used in automotive cooling systems.

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[20] E. G. Kimmich, "Rubber in Compression," ASTM BULLETIN, No. 106, October, 1940, p. 9.

[21] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 332.

Attention of the technical committee has been called from time to time to specific needs for various tests not yet standardized. These are being given consideration or have been referred to various other subcommittees.

Subcommittee VI on Packings (William Staniar, chairman).—Subcommittee VI has this year instituted the policy of having at its meetings a series of technical papers. The first program given at its March, 1941, meeting consisted of the following:

> "Experiences in Packing a Cellophane Casting Machine," by William Staniar,
> "Special Gaskets," by J. R. Boyer,
> "Tests of Packings for Refinery Services," by George W. Oxley, and
> "The Effect of Operating Variables on the Performance of a Typical Centrifugal Pump Packing," by F. C. Thorn.

These papers were exceedingly valuable, and it is expected that similar programs will be continued in the future. The subcommittee plans to give attention to the development of test methods applicable to automotive gaskets.

Subcommittee XI on Chemical Analysis of Rubber Products (S. Collier, chairman).—Since the completion of the revised methods of chemical analysis (D 297 - 40 T),[13] which were accepted by the Society through Committee E-10 on Standards, this committee has been actively at work on cooperative test programs dealing with the methods for determining cellulose and carbon black in rubber compounds. Work is also under way on Procedure B, rubber solvent method, looking toward its revision and improvement.

Subcommittee XIV on Abrasion Tests for Rubber Products (G. S. Haslam, chairman).—In addition to working on the proposed method for determining tear resistance, this subcommittee is undertaking a cooperative study of Methods A and B of the Standard Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 - 40). This work is being done jointly by a section of Subcommittee XIV and a committee organized by the Technical Committee of the Rubber Manufacturers Association. It is hoped by means of the program which has been developed, to obtain information concerning the reproducibility of Method B, using the National Bureau of Standards abrader, similar to that which was obtained by the committee with the cooperation of the V. L. Smithers Laboratories on Method A using the E. I. du Pont abrader. Effort will also be made to ascertain any possible correlation of the two methods. Another section of the subcommittee has been authorized to investigate possible methods for abrasion testing applicable to rubber and plastic decking materials such as rubber latex-cement flooring, vulcanized and cemented rubber matting, and the like, for which the present standard methods have been found unsuitable. This work is being undertaken at the suggestion of the Navy Department.

Subcommittee XVIII on Dynamic Fatigue Testing (W. F. Tuley, chairman).—The principal work before this subcommittee at present deals with the new Tentative Methods of Test for Compression Fatigue which are recommended for publication as tentative as mentioned earlier in this report. A cooperative investigation of the reproducibility of these tests in various laboratories is planned.

Subcommittee XV on Life Tests for Rubber Products (J. H. Ingmanson, chairman).—This subcommittee has under way a series of tests, in which eleven laboratories are cooperating, which is designed to investigate further the tentative revision of the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 – 40),[8] in which 80 C. is used and to compare the results obtained using this temperature with those at 70 C. The subcommittee is also undertaking further work on the Proposed Method of Test for Resistance to Accelerated Light Aging of Rubber Compounds[2] and the Proposed Method of Calibrating a Light Source for Accelerating the Deterioration of Rubber.[2] This work involves comparison of various types of artificial light sources and exposure cycles together with actual sunlight exposure.

Subcommittee XXIII on Hard Rubber (W. H. Juve, chairman).—A section of this subcommittee, organized under the chairmanship of W. E. Glancy for the preparation of standard test methods for asphalt composition battery boxes, has prepared proposed standard methods covering chemical examination, including manganese and iron determination, and acid absorption as well as physical tests for tensile strength, elongation, bulge tests, and sensitivity to hot and cold cycles. A large amount of cooperative testing has been done by the section, and these methods represent the results of the tests. It is expected that the methods will be ready for presentation to the Society through Committee E-10 later in the year.[13] The Section on Physical Testing under D. E. Jones has completed the testing program discussed in the 1940 report. The data from twelve cooperating laboratories have been tabulated and studied by the entire committee. The results indicated that the reproducibility of the present Tentative Methods of Testing Hard Rubber Products (D 530 – 39 T) is not all that could be desired, and further tests under more carefully controlled conditions are being carried out with the objective of improving these methods.

This report has been submitted to letter ballot of the committee which consists of 117 members; 75 members returned their ballots, of whom 67 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

O. M. HAYDEN,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-11 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

Testing Asphalt Composition Battery Containers (D 639 – 41 T),
Sampling and Testing Rubber Latex (D 640 – 41 T), and

Revision of Tentative Methods of:

Chemical Analysis of Rubber Products (D 297 – 40 T).

These recommendations were accepted by Committee E-10 on August 25, 1941, and the new tentative standards, assigned the A.S.T.M. serial designations indicated above, and the revised tentative methods appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 519, 509, and 466.

Committee D-11 also presented to the Society, through Committee E-10, Emergency Alternate Provisions in the Standard Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 41) which were accepted on August 25, 1941. They have been issued under the designation EA-D 27 and appear as an insert sheet in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 102.

REPORT OF COMMITTEE D-12

ON

SOAPS AND OTHER DETERGENTS

Committee D-12 on Soaps and Other Detergents held one meeting this year on March 11 and 12, 1941, in New York, N. Y. There was a larger attendance this year than there has been at previous sessions, when two meetings a year were held.

At this meeting, W. D. Pohle, of the Naval Stores Research Division of the U. S. Department of Agricultural Chemistry and Engineering, presented an informal talk on rosin soaps and their germicidal effects, and L. M. Diller, of Hellige-Diller, Inc., discussed the use of his photometer in reading the colors of liquid oils.

The following new members have been elected to Committee D-12 during the year:

T. W. Smith, U. S. Army Quartermaster Corps,
J. N. Borglin, Hercules Powder Co., Experiment Station,
B. G. Wilkes, Carbide and Carbon Chemical Corp.,
J. R. Donlan, Standard Alcohol Co.,
Chas. J. Gundel, Fels and Co., and
W. B. Sherry, General Chemical Co.

In addition, R. E. Divine, a former advisory member, has succeeded L. B. Hitchcock as a regular member and has also been elected to membership on the Advisory Committee in his place; Mr. Hitchcock, who has been a very active member of the committee and advisory board, resigned due to a change in duties. Bernice Bronner has replaced Katherine Fisher who resigned due to a change in duties.

Constructive criticism of the methods of test for soaps and detergents has been received from numerous sources, and these suggestions have been considered by the proper subcommittees. Some of the criticisms have been that the methods are too long, but the committee feels that as referee methods they must be accurate and precise, so that analysts somewhat inexperienced in soap work can obtain correct results. Any chemist on routine work can devise and use many short cuts on such work, but in the opinion of the committee, in referee methods accuracy is paramount.

The committee has worked in collaboration with purchasers in setting up specifications, particularly the Commonwealth of Pennsylvania.

A paper on "Soaps and Other Detergents" prepared by H. P. Trevithick with the assistance of a number of members of the committee, was published in the ASTM BULLETIN.[1] A summary of this paper appeared in *Industrial Standardization*, March, 1941, published by the American Standards Association; and in *American Dyestuff Reporter*, May, 1941. A somewhat similar article was also published in *Chemical Industries* in February, 1941; and in *Soap and Sanitary Chemicals*, July, 1941.

Considerable interest has been evinced in detergency measurements at various times. Section E on Sulfonated Detergents of Subcommittee II has worked on this problem for the past five years, and while the section has achieved considerable progress, it has not yet solved this problem. A summary of this work is in preparation and will be presented later.

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[1] ASTM BULLETIN, No. 107, December, 1940, p. 9.

I. NEW TENTATIVE STANDARDS

The committee recommends that the following new specifications and method of test be accepted for publication as tentative:

Specifications for Olive Oil Chip Soap (Type A, Pure; Type B, Blended),[2] and

Method of Test for Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution-Volumetric Method),[2] to be added, when adopted to the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 39).[3]

II. REVISIONS OF TENTATIVE STANDARDS

The committee recommends that the following tentative standards be revised as indicated below and continued as tentative:

Tentative Specifications for Palm Oil Chip Soap (D 536 - 39 T),[4] revised as appended hereto[5] to cover the addition of requirements for palm oil chip soap (blended type).

Tentative Specifications for Olive Oil Solid Soap (Type A, Pure; Type B, Blended (D 592 - 40 T),[6] revised as follows:

Table I.—Change the titer requirement for type A soap from "18 to 25 C." to read "16 to 26 C."

Add the following requirements for free acid as an editorial correction as these had been inadvertently omitted from the 1940 printing of the specifications:

	Type A	Type B
Free acid, calculated as oleic acid, max., per cent.......	0.2	0.2

Tentative Methods of Sampling and Chemical Analysis of Special Detergents (D 501 - 40 T),[7] revised by the addition of new Sections 13 to 15 covering a new method for sampling and analysis of tetrasodium pyrophosphate as follows:

13. *Sampling.*—Tetrasodium pyrophosphate ($Na_4P_2O_7$) shall be sampled by removing portions from various parts of the container. Samples shall not be taken from those portions where caking is noticeable due to the absorption of moisture and CO_2 through the container. If the tetrasodium pyrophosphate is caked, the sample shall be obtained by thoroughly mixing and quartering the entire contents of the package.

14. *Tetrasodium Pyrophosphate, $Na_4P_2O_7$*: (*a*) *Scope.*—This procedure describes an indirect determination of $Na_4P_2O_7$ by titration of H_2SO_4 liberated by the action of $ZnSO_4$ on an acid pyrophosphate in accordance with the following reactions:

$$Na_4P_2O_7 + 2HCl = Na_2H_2P_2O_7 + 2NaCl$$

$$Na_2H_2P_2O_7 + 2ZnSO_4 = Zn_2P_2O_7 + Na_2SO_4 + H_2SO_4$$

This titration is a measure of the pyrophosphate content. This method for pyrophosphate is inaccurate in the presence of polyphosphates.

(*b*) *Apparatus.*—Carry out the titration with an electrometric titration apparatus consisting of a potentiometer and glass electrode assembly.

(*c*) *Reagents:*

(*1*) *Hydrochloric Acid (0.2 N).*

(*2*) *Sodium Hydroxide Solution (0.2 N).*—Standardize against c.p. $Na_4P_2O_7$ which has been recrystallized three times from water and dried at 400 C. to constant weight.

(*3*) *Zinc Sulfate Solution.*—Dissolve 125 g. of $ZnSO_4 \cdot 7H_2O$ in distilled water and dilute to 1 liter, filter, and adjust the pH to 3.8.

(*d*) *Procedure.*—Weigh accurately approximately 1 g. of the sample and dissolve in sufficient distilled water in a 250-ml. beaker so that the resulting solution will just cover the electrodes of the glass electrode assembly. Adjust the pH of the solution to exactly 3.8 with 0.2 *N* HCl. Add 50 ml. of $ZnSO_4$ solution and allow 5 min. for the reaction to become complete as shown by the pH becoming constant. Titrate the liberated acid with 0.2 *N* NaOH until a pH of 3.8 is again reached.

(*e*) *Calculation.*—Calculate the percentage of tetrasodium pyrophosphate as follows:

$$Na_4P_2O_7\text{, per cent} = \frac{A \times F \times 100}{W}$$

where:

A = milliliters of NaOH required to titrate the sample,

F = grams of $Na_4P_2O_7$ equivalent to 1 ml. of 0.2 *N* NaOH solution used for titration as calculated by standardization against c.p. $Na_4P_2O_7$, and

W = weight of sample in grams.

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[2] These specifications and method were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 604 and 608, respectively.

[3] 1939 Book of A.S.T.M. Standards, Part III, p. 487.

[4] 1939 Book of A.S.T.M. Standards, Part III, p. 1063.

[5] These revised specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 606.

[6] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 508.

[7] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 516.

15. *Matter Insoluble in Water.*—Weigh 100 g. of the sample to the nearest 0.5 g. and transfer to a 1-liter beaker. Dissolve by stirring with distilled water at room temperature and dilute to approximately 900 ml. Filter by suction through a prepared, dried, and weighed Gooch crucible, using on the crucible a pad made of asbestos fiber only. Wash the beaker and residue free from alkali with water, and dry the crucible to constant weight in an oven at 110 C. Cool in a desiccator, and weigh. Calculate the percentage of matter insoluble in water from the average gain in weight of two checking duplicate determinations as follows:

Matter insoluble in water, per cent
= wt. of residue in grams.

Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 - 40 T),[8] the revision as appended hereto[9] includes revisions in seven definitions (alkali detergent, anhydrous soap content, blended soap, built soap, detergent, pure soap, soap powder), the omission of the definitions for detergent soap powder, soapy alkaline detergent, and the addition of new definitions for dry cleaning, scouring, and soil.

III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 39),[3] and accordingly asks for a favorable nine-tenths vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Section 12.—Change from its present form to read as follows to cover the addition of toluene as an alternate solvent for use in determining moisture in soaps containing 1 per cent or more of glycerol:

> 12. *Xylene (or Toluene).*—(For soaps containing 1 per cent or more of glycerol, toluene shall be used instead of xylene.) Saturate the xylene (or toluene) with water by shaking with a small quantity of water and distill. Use the distillate for the determination.

Section 13.—Following each reference to "xylene" add the words "or toluene" as a parenthetical phrase.

Section 14.—Change to read as follows by the addition of the italicized words:

> 14. Ethyl Alcohol (95 per cent).—Freshly boiled ethyl alcohol, 95 per cent or higher and neutral to phenolphthalein; *either Formula No. 3-A or No. 30 of the U. S. Internal Revenue Bureau.*

Section 29.—Omit this section covering the modified Wolff method for rosin, since the McNicoll method described in Section 30 is very satisfactory and a much more rapid procedure.

Section 32.—Add in Note 1, following this section, the present description of the Liebermann-Storch Test now appearing as a note at the end of Section 29.

Section 39.—Change the first eight sentences to read as follows by the addition of the italicized words and the omission of those in brackets:

> Weigh accurately from 0.10 to 0.50 g. (depending on the iodine number) of the fatty acids as prepared under Section 34 into a clean, dry, 450-ml. (16-oz.) glass-stoppered bottle containing 15 to 20 ml. of carbon tetrachloride. [or chloroform.] Add 25 ml. of the iodine solution from a pipette, allowing it to drain for a definite time. The excess of iodine should be from 50 to 60 per cent of the amount added, that is, from 100 to 150 per cent of the amount absorbed. [Moisten the stopper with KI solution (15 per cent) to prevent loss of iodine or chlorine, but guard against an amount sufficient to run down inside the bottle.] Let the bottle stand in a dark place for 30 min. [at a uniform temperature] *at a temperature of 25 ± 2 C.*; then add 20 ml. of KI solution (15 per cent) and 100 ml. of distilled water. Titrate the iodine with 0.1 *N* sodium thiosulfate, added gradually while shaking constantly, until the yellow color of the solution has almost disappeared. Add a few drops of starch paste and continue titration until the blue color has entirely disappeared. Toward the end of the reaction, stopper the bottle and shake vigorously, so that any iodine remaining in solution in the tetrachloride [or chloroform] may be taken up by the KI solution.

Section 42 (c).—Change the first sentence to read as follows by the addition of the italicized figure and the omission of the figure in brackets:

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[8] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 521.

[9] These revised definitions were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 617

> Evaporate the acidified solution (washing off and removing the cover glass, if used) to dryness on a steam bath or hot plate at a temperature not exceeding [200 C.] *120 C.*

Section 43.—In view of the publication as tentative of the new evolution-volumetric method for determination of carbon dioxide as an alternative procedure for the present standard train-absorption procedure, change this section from its present form to read as follows:

> 43. Whenever the determination of carbon dioxide is prescribed in the specifications, either the train-absorption method as described in Sections 44 and 45 or the evolution-volumetric method as described in the Tentative Method of Test for Carbon Dioxide in Soaps and Soap Products (A.S.T.M. Designation: D 460) of the American Society for Testing Materials,[2] shall be used.

Section 45.—Change the fifth sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> Weigh [out 1 or 2 g. of the sample] *a sufficient portion of the sample to yield approximately 0.2 g. of* CO_2 into the Erlenmeyer flask, cover with 20 ml. of freshly boiled distilled water, and close the apparatus with the train in place.

Add the following sentence at the end of this section:

> If potassium carbonate only is present, the increase in weight multiplied by 3.14 equals K_2CO_3.

Section 48 (c).—Change the second sentence to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> Dry, burn first at low heat,[a] and then ignite to constant weight, preferably in an electric furnace, at [950 to 1000 C.] *1050 to 1100 C.*

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[a] A platinum dish should not be used.

Section 53 (a).—Change the second sentence from its present form: namely,

> Transfer the insoluble matter, without drying, to a beaker and heat for 2.5 hr. with 200 ml. of water and 20 ml. of HCl (sp. gr. 1.125) in a flask provided with a reflux condenser.

to read as follows:

> Transfer the insoluble matter, without drying, to a 500-ml. flask provided with a reflux condenser, add 20 ml. of HCl (sp. gr. 1.125) and 200 ml. of water, and boil for 2.5 hr.

Section 59 (b).—Change the chemical formulas "K_2PtCl_6" to read "H_2PtCl_6."

IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative standards be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for:

- Built Soap, Powdered (D 533 - 39 T),[10]
- Soap Powder (Alkaline Soap Powder) (D 534 - 39 T),[10]
- Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D535 - 40 T),[11]
- Sodium Metasilicate (D 537 - 39 T),[10] and
- Sodium Sesquisilicate (D 594 - 40 T).[11]

Tentative Methods of:

- Chemical Analysis of Sulfonated and Sulfated Oils (D 500 - 38 T).[10]

V. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

The committee recommends that the revision[12] of the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 39)[3] published as tentative in 1940 comprising a revision of the procedure for determination of free alkali, matter insoluble in water, and the titer test and titer test thermometer be approved for reference to letter ballot of the Society for adoption as standard.

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[10] 1939 Book of A.S.T.M. Standards, Part III, pp. 1065, 1067, 1068, 1074.

[11] 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 510, 514.

[12] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 573.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 70 members; 56 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (J. B. Crowe, chairman):

Section A on Soaps (M. L. Sheely, chairman) recommends that the revision of the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 39) published as tentative last year be adopted as standard. This revision covers minor changes in the procedure for determining free alkali, omitting the filtration if the alkaline builder is under 0.5 per cent; in the determination of water insoluble matter; and in the titer test. The committee is recommending for immediate adoption a revision in Method D 460 consisting of the elimination of the Wolff method for rosin and the use of toluene in the moisture test if 1 per cent or more of glycerol is present. In addition, editorial changes are made in those sections of the methods covering carbon dioxide, starch, and potash.

The section is now recommending that the proposed Method of Test for Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution-Volumetric Method), as appended hereto,[2] be accepted for publication as tentative, to be added when adopted to Standard Methods D 460 – 39.[3]

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Olive Oil Chip Soap (Type A, Pure; Type B, Blended)	50	0	6
Test for Carbonates as Carbon Dioxide in Soaps and Soap Products (Evolution-Volumetric Method)	50	0	6
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Palm Oil Chip Soap (D 536 – 39 T)	50	0	6
Spec. for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 – 40 T)	50	0	6
Sampling and Chemical Analysis of Special Detergents (D 501 – 40 T)	55	0	1
Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 40 T)	54	1	1
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 39)	54	0	2
IV. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Spec. for Built Soap, Powdered (D 533 – 39 T)	55	0	1
Spec. for Soap Powder (Alkaline Soap Powder) (D 534 – 39 T)	55	0	1
Spec. for Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 – 40 T)	55	0	1
Spec. for Sodium Metasilicate (D 537 – 39 T)	55	0	1
Spec. for Sodium Sesquisilicate (D 594 – 40 T)	55	0	1
Chemical Analysis of Sulfonated and Sulfated Oils (D 500 – 38 T)	55	0	1
V. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 39)	55	0	1

Through the cooperation of this committee with Technical Committee XII on Laboratory Apparatus of A.S.T.M. Committee E-1 on Methods of Testing and the American Oil Chemists Society, Fat Analysis Committee, detailed revised specifications for the titer test thermometer[13] have been completed. The Association of Official Agricultural Chemists is also considering the adoption of this titer test thermometer. It is expected that the specifications for this thermometer will be acceptable to the several interested groups in the interest of having a single standard for this thermometer.

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[13] See Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials, 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 619.

Section B on Sulfonated Detergents (Ralph Hart, chairman) is working on new methods for volatile solvents in sulfonated oils and hopes to be able to complete this work for submission at the next meeting.

Section C on Dry Cleaning Detergents of this subcommittee and also Section D of Subcommittee II (G. P. Fulton, chairman) are investigating performance tests and specifications and hope to achieve some concrete results by the next meeting.

Section D on Special Detergents (W. H. Koch, chairman) carried out cooperative laboratory work on a method of analysis for tetrasodium pyrophosphate. This method consisted of a revision, where necessary, of the method by which the committee members carried out similar studies during the previous year. The work consisted of investigations directed to decide which of two methods of preparation of c.p. tetrasodium pyrophosphate would provide a purer product for purposes of standardization. This was followed by the analyses of two commercial products for $Na_4P_2O_7$ content. The agreement between the results obtained by the eleven laboratories supplying analyses was exceptionally good. The individual results obtained by each laboratory were excellent. The section feels that the new procedure presented for inclusion in the Tentative Methods of Sampling and Chemical Analysis of Special Detergents (D 501 – 40 T)[7] is more than adequate for the purpose intended. The committee as a whole has approved the recommendation of this subcommittee to submit the method as tentative.

A method of analysis for sodium orthosilicate was also considered, but it was decided to withhold this method until it was shown that there was considerably more demand for it and until certain highly controversial arguments on chemical composition were more completely clarified. Further progress on this method will await the action of the Subcommittee on Specifications.

Subcommittee II on Specifications (F. W. Smither, chairman):

This subcommittee held one meeting during the year. The various sections of Subcommittee II have been active, but the time and study required for the difficult assignments now under consideration have necessarily limited the number of definite recommendations at this time. Brief mention is made below of several recommendations on new and revised specifications which are covered in detail earlier in this report.

Section A on Textile Soaps (C. L. Nutting, chairman) and Section C on Straight Soaps (Israel Katz, chairman) have continued their joint work as reported last year. It is recommended that the Tenative Specifications for Palm Oil Solid Soap (Type A, Pure; Type B, Blended) (D 535 – 40 T) be adopted as standard. It is also recommended that the Tentative Specifications for Palm Oil Chip Soap (D 536 – 39 T) be revised to include two types, pure and blended, and continued as tentative. Revisions are being recommended in the titer values and the fatty acid limit of the Tentative Specifications for Olive Oil Solid Soap (Type A, Pure; Type B, Blended) (D 592 – 40 T) and the specifications continued as tentative. Proposed Specifications for Olive Oil Chip Soap (Type A, Pure; Type B, Blended)[2] are being submitted for acceptance.

These sections will work on "Fig" (potash) soaps and liquid soaps this year.

Section B on Built Soaps (Frederick Krassner, chairman) is recommending that the Tentative Specifications for Built Soap, Powdered (D 533 – 39 T) and for Soap Powder (Alkaline Soap Powder) (D 534 – 39 T) be adopted as standard. The section is also recommending that the Tentative Specifications for Salt Water Soap (D 593 – 40 T) be continued as tentative. The section has under consideration the formulation of specifications for scouring powders and grit soaps.

Section D on Dry Cleaning Detergents (G. P. Fulton, chairman) reports that information is not available at this time for preparing specifications for dry-cleaning soaps, based on composition or on physical characteristics. This section will make a survey of the producers, users, and testing laboratories by means of a questionnaire for the purpose of determining the practicability of preparing performance specifications.

Section E on Sulfonated Detergents (J. B. Crowe, chairman) has prepared a résumé of, and detergency tests for, all of the previous work which has been done by this section. These data are now compiled in condensed form which is to be subjected to statistical study by the section members. Also, a questionnaire is to be sent out covering such points as history and performance of standard soil fabrics.

Section F on Special Detergents (C. C. Zeigler, chairman) is recommending that the Tentative Specifications for Sodium Sesquisilicate (D 594 – 40 T) and for Sodium Metasilicate (D 537 – 39 T) be adopted as standard and that the Tentative Specifications for Tetrasodium Pyrophosphate (Anhydrous) (D 595 – 40 T) be continued as tentative. The section is considering the preparation of specifications for sodium orthosilicate.

Section G on Metal Cleaners (J. C. Harris, chairman) is preparing methods of analysis for metal cleaners, and it is expected that the methods will be completed for submission to the section for approval for presentation as a tentative standard at the 1942 annual meeting. The section plans to circulate a questionnaire among producers and consumers of metal cleaning compositions for the purpose of ascertaining what standards of cleanliness should be prescribed and what methods of determining this property may be employed. It is believed that the results of this questionnaire will enable the section to formulate useful specifications and methods for the metal cleaning industry.

Subcommittee III on Nomenclature and Definitions (C. A. Marlies, chairman):

This subcommittee recommends that the Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 40 T) be revised as mentioned earlier in this report and continued as tentative.

The subcommittee also recommends that the title of the Tentative Specifications for Soap Powder (Alkaline Soap Powder) (D 534 – 39 T) be changed editorially, since the subcommittee feels that the subtitle "Alkaline Soap Powder" is more appropriate and descriptive than the present main title "Soap Powder." In this way it is hoped to educate the industry to a better designation of this time-honored product.

Subcommittee IV on Papers and Publications (W. H. Koch, chairman):

The paper on "Soaps and Other Detergents," by H. P. Trevithick, which was approved last year was published in the ASTM BULLETIN[1] as mentioned earlier in this report.

The subcommittee prepared the two sections on soda ash and trisodium phosphate for a monograph on the testing and use of soaps and other detergents. These sections were submitted to the committee during the year with the request for their comments. Many were received, and the committee was considering the desired additions and corrections when the question of the desirability of presenting such a monograph again arose. This question will again be put to all the members of the committee by letter with a request for a definite expression of opinion by each person. One suggestion is that the monograph be in the form of a tabulation with explanation of such things as pH at various temperatures and concentrations, total alkalinity to methyl orange, commercial forms, common names, package sizes, common uses in the detergent field, solubility at 20 C., relative rates of solution, colloidal and emulsifying properties, surface tension, stability in storage, corrosion rates against metals, paints, fabrics, etc., water softening properties, turbidity, sudsing in combination with soap, oil emulsification, action on the skin, and other properties which the committee might include later, for each of the separate detergents for which the committee may have provided specifications and methods of analysis.

It was also suggested and recommended that Subcommittee IV prepare for publication and presentation to the Society the method of analysis for tetrasodium pyrophosphate which had been developed by Section D of Subcommittee I. It was felt that the type of work this section has done, in this instance in their cooperative laboratory work, is of scientific value and should be made available to the profession. Accordingly, this method has been prepared for inclusion in the Tentative Methods of Sampling and Chemical Analysis of Special Detergents (D 501 - 40 T) as recommended earlier in this report.

This report has been submitted to letter ballot of the committee which consists of 70 members, 56 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. P. TREVITHICK,
Chairman.

B. S. VAN ZILE,
Secretary.

REPORT OF COMMITTEE D-13
ON
TEXTILE MATERIALS

Committee D-13 on Textile Materials held two meetings during the year: one on October 16 to 18, 1940, in New York, N. Y., and the other on March 5 to 7, 1941, in Washington, D. C. The latter meeting was held in conjunction with the spring meeting of the Society. The number of members and guests registered was 153 and 136, respectively. The total attendance at these two meetings set a new high record. Of the 28 subcommittees or sections which compose the organization of Committee D-13, 24 held sessions at the fall meeting and 19 at the spring meeting. The executive direction of the affairs of the committee required three meetings of the officers and two meetings of the Advisory Committee.

The above facts, together with the numerous subcommittee activities and the many recommendations regarding standards reported herein, indicate that interest in standardization in the textile field is fully as active and productive as in recent years. The officers of the committee take this opportunity to express their full appreciation of the hearty and voluntary service rendered by the chairmen and members of the subcommittees during the past year.

The Advisory Committee has approved of one change in its membership. Mr. A. C. Clifford has been appointed chairman of Section I on Nomenclature and Definitions, of Subcommittee A-8 on Glass Fiber and Its Products, *vice* S. M. Dockerty, resigned. The Advisory Committee has also appointed W. D. Appel to represent Committee D-13 on Technical Committee XIII on Hydrogen Ion Determinations, of Committee E-1 on Methods of Testing.

Mr. A. G. Ashcroft has accepted an appointment to the Committee on Application of Statistical Methods to Quality Control of Materials and Manufactured Products, organized by the American Standards Association at the request of the War Department. By action of the Executive Committee of the Society, C. J. Huber and F. S. Mapes have been named as nonvoting delegates of the American Society for Testing Materials on the Inter-Society Color Council, and H. J. Ball has been appointed as the A.S.T.M. representative on the Advisory Committee on Ultimate Consumer Goods of the American Standards Association. Messrs. A. G. Ashcroft, W. M. Scott, and R. E. Hess (Alternate) have been appointed as representatives of the A.S.T.M. on the recently organized Sectional Committee on Fastness of Colored Textiles, of which the Society is co-sponsor, functioning under the procedure of the American Standards Association.

Mr. L. B. Arnold, Jr., has resigned as chairman of the Special Committee for testing of Textile Finishes, because of a change of position, and M. C. Moore and G. A. Slowinske have been appointed chairman and vice-chairman, respectively, by the sponsors of the program. A report of the work for the second year on this project is given under the activities of the subcommittees.

The committee feels it is an honor to place in its report a record of the nomination of one of its very active members, Dean Harvey, to the office of vice-president of the Society.

The American Standards Association has approved as an American standard the A.S.T.M. Standard Methods of Testing and Tolerances for Tubular Sleeving and Braids (D 354 – 36; ASA L 13 - 1941). The committee is recommending to the ASA for approval as American standard two other standards: namely, the Standard Specifications for Textile Testing Machines (D 76 - 40) and the Standard Methods of Testing Wool Felt (D 461 - 40).

The Advisory Committee authorized at the October meeting the formation of a Membership Committee. Its function is to seek out and encourage the enrollment of individuals who, because of their interests, experience, and attitude, can contribute to the work of Committee D-13. The Membership Committee will also concern itself with an analysis of those features of the work of Committee D-13 which make it an attractive field for the efforts of those interested in standardization in the textile industry and, if they exist, of any features of the organization or methods of operation which might discourage the whole-hearted cooperation of those who could otherwise contribute to the committee's progress. The Membership Committee is composed of G. E. Hopkins, chairman, K. B. Cook, and D. G. Woolf. It has already approached 109 prospective members and has secured 23 new members.

Acting upon the request of Subcommittee B-4 on Bleaching, Dyeing, and Finishing (W. M. Scott, chairman), Committee D-13 voted at the March meeting to authorize the appointment of a Special Committee on Dermatitis. This committee will advise with similar committees from several other national associations, which have a kindred interest in this subject, upon the possibility of joint action in formulating a program of research into textile finishes as a cause of skin irritation. The committee is composed of Ephraim Freedman, chairman, W. H. Cady, C. W. Dorn, D. H. Powers, W. M. Scott, and J. H. Shipp.

It is with regret that the committee records the decease of Edwin H. Marble at his home in Worcester, Mass., on April 16, 1941. He was a charter member of the committee. Mr. Marble was intimately associated with the formation of Committee D-13, and his advice and wise counsel were of valuable assistance in guiding it through its early and formative years.

The present membership of Committee D-13 is 239; of whom 103 are classified as producers, 53 as consumers, and 83 as general interest members.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication as tentative two new specifications, five new methods of test for textile materials, and is recommending the revision of seven tentative standards, the revision of four existing standards for immediate adoption, the tentative revision of six existing standards, the adoption as standard of three tentative standards and of three tentative revisions of existing standards, and the withdrawal of two existing standards. The revisions being presented are included in detail in Appendix I and are accompanied, where necessary, by brief statements of the reasons for such revisions. The new tentative standards and the existing standards affected are listed in Table I and are also referred to under subcommittee activities.

These recommendations have been submitted to letter ballot of the committee which consists of 239 members, the results being shown in Table I.

Subsequent to the 1940 annual meeting, Committee D-13 presented to the Society through Committee E-10 on Standards an extensive revision of the Tentative General Methods of Testing Cotton Fibers (D 414 – 37 T). The recommendation was accepted[1] by Committee E-10 on August 28, 1940, and the revised methods now appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 446, under the designation D 414 – 40 T.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Spec. for Medium-Weight Cotton Corduroy Fabrics	29	1	68
Spec. for Fire-Retardant Properties of Treated Textile Fabrics	46	1	51
Tests for Evaluating Compounds Designed to Increase the Resistance of Textiles to Insect Pests	45	2	51
Testing Asbestos Tubular Sleeving	20	0	78
Quantitative Analysis of Textiles	69	0	29
Test for Commercial Weight of Continuous Filament Rayon Yarns (D 258 – 41 T)	37	1	60
Test for Commercial Weight of Spun Rayon Yarns and Threads (D 507 – 41 T)	38	1	59
II. REVISIONS OF TENTATIVE STANDARDS			
Spec. for Single-Ply Bleached Cotton Broadcloth (D 504 – 38 T)	37	1	60
Testing and Tolerances for Woven Glass Fabrics (D 579 – 40 T)	21	0	77
Testing and Tolerances for Woven Glass Tapes (D 580 – 40 T)	21	0	77
Testing Rayon Staple (D 540 – 39 T)	36	0	62
Test for Resistance of Textile Fabrics and Yarns to Moths (D 582 – 40 T)	49	2	47
Test for Shrinkage of Grease Wool (Laboratory Scale Operations) (D 584 – 40 T)	37	0	61
Identification of Fibers in Textiles and Quantitative Analysis of Textiles (D 276 – 37 T)	71	0	27
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Testing and Tolerances for Cotton Yarns (D 180 – 40)	48	1	49
Testing and Tolerances for Tubular Sleeving and Braids (D 354 – 36; ASA L13-1941)	22	0	76
Test for Fastness of Dyed or Printed Cotton Fabrics to Laundering or Domestic Washing (D 435 – 37)	47	0	51
Test for Fastness of Colored Textile Fabrics to Light (D 506 – 39)	53	2	43
IV. TENTATIVE REVISIONS OF STANDARDS			
Spec. for Textile Testing Machines (D 76 – 40)	63	0	35
Spec. and Test for Fineness of Wool Tops (D 472 – 41)	34	0	64
Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 38)	34	1	63
Testing and Tolerances for Cotton Yarns (D 180 – 40)	43	1	54
Testing Pile Floor Covering (D 418 – 40)	39	0	60
Definitions of Terms Relating to Textile Materials (D 123 – 40)	70	0	28
V. ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Testing and Tolerances for Single Jute Yarn (D 541 – 39 T), as revised	67	0	64
Spec. and Test for Fineness of Wool Tops (D 472 – 40 T)	33	0	65
Definitions of Terms Relating to Textile Materials (D 123 - 40 T), as revised	71	0	27
VI. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARD			
Spec. for Textile Testing Machines (D 76 – 40)	63	0	35
Spec. and Test for Asbestos Tape for Electrical Purposes (D 315 – 37)	19	0	79
Spec. and Test for Asbestos Roving for Electrical Purposes (D 375 – 37)	20	0	78
VII. WITHDRAWAL OF STANDARDS			
Spec. and Test for Asbestos Yarns (D 299 – 37)	29	0	69
Spec. and Test for Cuban (Jute) Raw Sugar Bags (D 275 – 33)	33	0	65

ACTIVITIES OF SUBCOMMITTEES

The recommendations being submitted indicate that the various subcommittees and sections have been quite active during the year. Many important projects are under consideration and are mentioned briefly in the following summary of subcommittee activities.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee D-13 reported results of the letter ballot vote as follows: Of a total membership of 214, 131 members returned their ballots, of whom 72 voted affirmatively, 0 negatively, and 59 members marked their ballots "not voting."

Subcommittee A-1 on Cotton and Its Products (K. B. Cook, chairman):

Section I on Cotton (R. W. Webb, chairman) was responsible for the complete revision of the Tentative General Methods of Testing Cotton Fibers (D 414 – 37 T), mentioned earlier in this report.

Section II on Cotton Yarns and Threads (A. C. Clifford, chairman) is recommending for immediate adoption a revision of the Standard General Methods of Testing and Tolerances for Cotton Yarns (D 180 – 40), as given in Appendix I, which will substitute for "standard regain" a formally adopted "commercial regain" for use in checking shipping weights. Consequently, it also recommends the withdrawal of the revision of Section 2 (*c*) of Methods D 180 covering standard regain which was published as tentative in 1938 and modified in 1940.[2]

The section is recommending a revision for publication as tentative, of the methods of determining twist in Sections 11 (*a*) and (*b*) of Methods D 180, as given in Appendix I.

A tentative revision[2] of Methods D 180 covering a test for appearance has been published for three years. The plates have been modified this year and, therefore, the section recommends that the revision be continued as tentative.

The section is recommending the incorporation of the tentative revisions of Section 2 (*c*) relating to standard regain and Paragraphs A4 (*c*) and A5 in the Appendix to Methods D 180 as an editorial matter as given in Appendix I to this report, since this Appendix to the methods is published as information only.

Studies are being conducted on development of requirements for number of tests, amount of tension to be applied in twist determinations, and clamps for strength tests of heavy plied yarns.

Section III on Light and Medium Weight Cotton Woven Fabrics (R. T. Fisher, chairman) has been assigned jurisdiction over specifications and tolerances for fabrics made of spun rayon and mixtures of spun rayon and other fibers. Studies are being made of specifications for fine fancy goods, cotton and synthetic yarn mixtures, and fine staple gray goods.

The section is also working on a proposed revision of the Standard Specifications and Methods of Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 – 40) to include additional fabrics which, from the trend of the trade, seem desirable.

Section IV on Tire Fabrics (Martin Castricum, chairman) recommends that the revision of the Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 38), published as tentative in 1939 which provides for number of tests, be further revised, as given in Appendix I, to adjust the precision so that it is more in line with the character of tire cord tests.

The section is continuing its study of the comparative advantages of the conditioned and oven-dry methods of testing, and is considering the establishment of minimum color and foreign matter appearance standards for tire fabrics other than cord.

Section V on Heavy Cotton Woven Fabrics (B. L. Whittier, chairman) is continuing its study on moisture regain in different types of heavy cotton fabrics, and the elimination of jaw breaks in testing heavy fabrics for strength.

Section VI on Narrow Fabrics (F. S. Mapes, chairman) is recommending for immediate adoption a revision, as given in Appendix I, of the Standard Methods of Testing and Tolerances for Tubular Sleeving and Braids (D 354 – 36), covering the measurement of inside diameter and yards per pound.

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[2] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 571.

The section is recommending that the Tentative Methods of Testing and Tolerances for Woven Tapes (D 259 - 39 T) be continued as tentative pending the results of a study of means of measuring thickness.

The section is considering the inclusion of tapes other than 0.007-in. in the Standard Specifications for 0.007-in. Cotton Tape for Electrical Purposes (D 335 - 36).

Subcommittee A-2 on Rayon and Its Products (A. M. Tenney, chairman):

Section I on Rayon (A. M. Tenney, chairman) recommends for publication as tentative a revision of the Standard Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 40),[3] in the form of a new proposed Tentative Method of Test for Commercial Weight of Continuous Filament Rayon Yarns as appended hereto.[4]

The section is conducting interlaboratory tests on filament rayon yarns to establish statistically requirements for number of tests in Methods D 258.

Section II on Rayon Fabrics (Alexis Sommaripa, chairman) recommends that the Tentative Method of Test for Resistance to Yarn Slippage in Silk, Rayon, and Silk-Rayon Woven Fabrics (D 434 - 36 T) be continued as tentative, pending the results of its study of means to control shrinkage which has an important bearing on the results of the test for slippage.

The section is studying methods of testing the relative merits of rayon fabrics in actual consumer use.

Section III on Rayon Staple and Spun Rayon Yarn (K. B. Cook, chairman) recommends for publication as tentative a revision of the Standard General Methods of Testing and Tolerances for Spun Rayon Yarns and Threads (D 507 - 39),[5] in the form of a new proposed Tentative Method of Test for Commercial Weight of Spun Rayon Yarns and Threads as appended hereto.[6] The section is also recommending a revision in the Tentative Methods of Testing Rayon Staple (D 540 - 39 T) as given in Appendix I.

Subcommittee A-3 on Wool and Its Products (A. G. Ashcroft, chairman):

Section I on Wool (Werner von Bergen, chairman) recommends the adoption as standard of the Tentative Specifications and Methods of Test for Fineness of Wool Tops (D 472 - 40 T).[7] An addition to this standard covering requirements for wool top grades below 50's is being recommended for publication as tentative as given in Appendix I. This section is also recommending a revision of the Tentative Method of Test for Shrinkage of Grease Wool (Laboratory Scale Operations) (D 584 - 40 T) based on experience in its use by the U. S. Treasury Department and U. S. Department of Agriculture during the past year. Methods D 584 in their completely revised form are appended hereto.[8]

The section is studying mohair grades with a view to establishing specifications, a sampling tool for raw wool, determination of vegetable matter in wool, and moisture content in scoured wool.

Section II on Felt (G. H. Harnden, chairman) is studying the new S.A.E. Standards on Felt as a basis for A.S.T.M. standards on industrial felts. Methods for the determination of hardness, shrinkage, and swelling by water of felts are also being studied.

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 164.

[4] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 562.

[5] 1939 Book of A.S.T.M. Standards, Part III, p. 437.

[6] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 576.

[7] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 479.

[8] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 530.

Section IV on Pile Floor Coverings (J. N. Dow, chairman) is recommending for publication as tentative a revision of the Standard Methods of Testing Pile Floor Covering (D 418 – 40) to add a form for reporting pile floor covering analyses as given in Appendix I.

The section is developing a machine for wear testing of pile fabric and has available a machine for testing "shedding." The data from the tests to standardize machine wear tests and service tests are being analyzed. The section is also working on standards for normal service for use in mothproofing tests and method of test for effectiveness of fireproofing treatments.

Section V on Woolen and Worsted Fabrics (F. A. Prisley, chairman) is recommending that the Tentative Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 – 37 T) be continued as tentative pending the results of a study being made to bring the standard into conformity with the Wool Products Labelling Act of 1939.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman):

This subcommittee submits for publication as tentative new proposed Tentative Methods of Testing Asbestos Tubular Sleeving as appended hereto.[9] The subcommittee recommends the adoption as standard of the tentative revisions[10] of the Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 – 37)[11] and of the tentative revisions[10] of the Standard Specifications and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 – 37).[11] It is also recommending that the Standard Specifications and Methods of Test for Asbestos Yarns (D 299 – 37)[12] be withdrawn as they have been completely revised and published in the form of Tentative Specifications and Methods of Test for Asbestos Yarns (D 299 – 40 T). An editorial change is being made in Tentative Methods D 299 as given in Appendix I.

The subcommittee is studying methods for determining magnetic rating of asbestos fiber and textiles, resistivity and chemical methods for evaluation of asbestos textile products, and for total iron content. Specifications for fifteen weights and constructions of asbestos cloth acceptable to the industry as general standards are being developed. A research project on the relationship of the breaking strength characteristics of various grades of asbestos cloth to temperature and time of heating is to be undertaken if sufficient financial support is obtained.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (A. R. Howe, chairman):

This subcommittee is recommending for adoption as standard the Tentative Methods of Testing and Tolerances for Single Jute Yarn (D 541 – 39 T), revised as given in Appendix I. These methods have received the unqualified approval of all manufacturers represented in the Soft Fibre Manufacturers' Institute in addition to unanimous approval in the committee.

An extensive survey of the consumers of sugar bags shows that the majority consider the Standard Specifications and Methods of Test for Cuban (Jute) Raw Sugar Bags (D 275 – 33)[13] of no value or use to them. The subcommittee is, therefore, recommending the withdrawal of this standard.

The subcommittee is developing methods of test and tolerances for electrical yarn and roving made of jute.

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[9] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 552.

[10] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 571.

[11] 1939 Book of A.S.T.M. Standards, Part III, pp. 385, 391.

[12] 1939 Book of A.S.T.M. Standards, Part III, p. 388.

[13] *Ibid.*, p. 435.

Subcommittee A-6 on Household and Garment Fabrics (Ruth O'Brien, chairman):

This subcommittee is recommending for publication as tentative Specifications for Medium-Weight Cotton Corduroy Fabrics as appended hereto.[14] A revision of the tentative Specifications for Single-Ply Bleached Cotton Broadcloth (D 504 – 38 T)[15] is also recommended, and are appended hereto[16] in their completely revised form.

The subcommittee projects include methods of test to determine degree of mercerization; serviceability tests for bed sheeting and other household fabrics; specifications and test methods for outing flannel, dish toweling, blanketing, work garment, and upholstery fabrics. In collaboration with Section II on Rayon Fabrics, of Subcommittee A-2, a special committee is working on specifications for rayon dress fabrics.

Subcommittee A-8 on Glass Fiber and Its Products (Dean Harvey, chairman):

This subcommittee recommends revisions of the Tentative Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 – 40 T), and for Woven Glass Tapes (D 580 – 40 T), changing the ranges of nominal thickness for certain permissible variations in average thickness as given in Appendix I.

The subcommittee is studying methods of test for essential properties of sliver for wire insulation and for use as filler, determination of yarn number, essential properties of sizing (lubricant and binder) for glass yarns, and abrasion of glass yarns and fabrics.

Subcommittee B-1 on Methods and Machines (W. D. Appel, chairman):

Section I on Methods (W. D. Appel, chairman) recommends an extensive revision of the Tentative Methods for Identification of Fibers in Textiles and Quantitative Analysis of Textiles (D 276 – 37 T),[17] consisting of a complete rewriting of the methods for quantitative analysis. It is further recommended that the revised methods be deleted from Tentative Methods D 276 – 37 T and issued as new Tentative Methods of Quantitative Analysis of Textiles, as appended hereto.[18]

Since the section is working on a revision of the identification methods in Tentative Methods D 276 – 37 T, it therefore recommends this portion be continued as tentative.

The section is studying methods for resilience of fibers, resistance of textiles to soiling and to gas fading, and for accelerated aging of textiles. A joint committee with the American Association of Textile Chemists and Colorists is gathering data on fundamental properties of fibers.

Section II on Machines (H. J. Ball, chairman) recommends the adoption as standard of the tentative revision[19] in the Standard Specifications for Textile Testing Machines (D 76 – 40)[20] on calibration of thickness gages. This section is also recommending a tentative revision of Specifications D 76 to provide specifications for twist testers, as set forth in Appendix I.

The section is undertaking the development of specifications for yarn reels and drying ovens, and a study of the sensitivity of vertical Type A testing machines.

Section III on Atmospheric Conditions (R. H. Brown, chairman) is studying existing techniques for drying out tests to determine moisture regain, for conditioning samples to the standard atmosphere, and for calculating commercial regain of mixed materials, with a view to preparing recommended procedures.

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[14] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 555.

[15] 1939 Book of A.S.T.M. Standards, Part III, p. 979.

[16] These revised specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 553.

[17] 1939 Book of A.S.T.M. Standards, Part III, p. 951.

[18] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 537.

[19] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 570.

[20] *Ibid.*, p. 140.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman):

This subcommittee is recommending the adoption as standard of the Tentative Definitions of Terms Relating to Textile Materials (D 123 – 40 T) with the note which accompanies the definition of standard moisture regain and the definition of sliver revised as given in Appendix I. This subcommittee also recommends a tentative revision of the definition of commercial weight and that the definitions of the terms "commercial weight (rayon)" and "resilience" be accepted for publication as tentative as given in Appendix I. It is also recommended that a number of terms relating to the "hand" of fabrics be added to the glossary of definitions. These will appear in the 1941 edition of the compilation of "A.S.T.M. Standards on Textile Materials."

The subcommittee has prepared a new Recommended Practice for the Designation of Yarn Construction which will be published as information in the 1941 edition of "A.S.T.M. Standards on Textile Materials."

The subcommittee has a number of other definitions and revisions under consideration.

Subcommittee B-4 on Bleaching, Dyeing and Finishing (W. M. Scott, chairman):

This subcommittee is recommending for publication as tentative two new tentative standards: namely, Tentative Specifications for Fire-Retardant Properties of Treated Textile Fabrics[21] and Tentative Methods of Test for Evaluating Compounds Designed to Increase the Resistance of Fabrics and Yarns to Insect Pests[22] as appended hereto. The committee has also prepared proposed Methods for Identification of Finishes on Textiles (Qualitative) which are published as information as given in Appendix II.

The subcommittee also recommends certain revisions in the Tentative Method of Test for Resistance of Textile Fabrics and Yarns to Moths (D 582 – 40 T).[23] The revised method is appended hereto.[24]

The subcommittee is recommending that the Tentative Method of Test for Volumetric Determination of Small Amounts of Copper in Textiles (D 463 – 37 T) be continued as tentative without revision.

The subcommittee is recommending for immediate adoption revisions of the Standard Method of Test for Fastness of Dyed or Printed Cotton Textiles to Laundering or Domestic Washing (D 435 – 37) and the Standard Method of Test for Fastness of Colored Textile Fabrics to Light (D 506 – 39). These methods in their completely revised form are appended hereto.[25]

Acting on a recommendation of this subcommittee, the chairman of Committee D-13 has appointed a committee, as mentioned earlier in this report, to cooperate in the formulation of a research program on textile finishes as a cause of dermatitis.

Subcommittee B-5 on Sampling, Presentation, and Interpretation of Data (A. G. Ashcroft, chairman):

This subcommittee is actively continuing its cooperation with other subcommittees in making statistical studies of data with a view to establishing the number of tests required to give a desired precision. Some minor editorial changes are being made in the Proposed Recommended Practice for Calculating Number of Tests to be Specified in Determining Average Quality of a Textile Material which will again be published as information in the 1941 edition of the compilation of "A.S.T.M. Standards on Textile Materials."

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[21] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 526.

[22] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 548.

[23] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 422.

[24] This revised method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 546.

[25] These revised methods were adopted as standard by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 163 and 144, respectively.

Subcommittee B-6 on Papers (R. H. Brown, chairman):

This subcommittee arranged for the presentation of the following papers at the October, 1941, meeting (No papers session was held at the March, 1941, meeting):

"Comparison of the Breaking Strength of Fabrics as Determined by the Pendulum and Inclined Plane Testing Machines," by Gladys White and Emma C. Peterson,

"A Correlation Between Laboratory Abrasion Tests and the Service Life of Men's and Women's Outerwear," by Arthur Russman,

"The New Synthetic Textile Fibers," by H. R. Mauersberger, and

"Relative Merit of Three Rayon Taffetas in Consumer Service," by Alexis Sommaripa.

Abstracts of the above papers will appear in the 1941 edition of the compilation of "A.S.T.M. Standards on Textile Materials."

Special Committee for Testing of Textile Finishes (L. B. Arnold, Jr., chairman):

The work of this special committee received sufficient financial backing from industry to warrant its continuance for a second year. Edwin C. Dreby was continued as the research associate and S. W. Adams was engaged for a part of the year as his assistant.

The current phase of the program has been the development of instruments for the evaluation of the "hand" of fabrics, and satisfactory progress is reported. Three instruments, called the Planoflex, the Friction-Meter, and the Contour-Meter, have been designed, tested, and improved. Fabrics with various types of finish have been tested with them, and the results studied with respect to reproducibility, sensitivity, and as to agreement with expert's tactual evaluation and rating of the fabrics. The first two instruments detected not only differences which were appreciated by experts, but in many cases differences which were not observed by them. The differences corresponded to the known treatments which had been given the fabrics. The Contour-Meter has not proved to be equally sensitive.

The subscribers have appointed a committee to set up a program for a third year's work, and to determine whether financial backing can be secured for it. Another committee has as its function, first, to present to Committee D-13 for adoption instruments and methods which have been developed, second, to promote their general use and to answer inquiries which may arise regarding them, and third, to renew the research program at such time as may be propitious.

This report has been submitted to letter ballot of the committee which consists of 239 members; 97 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

APPENDIX I

PROPOSED REVISIONS IN STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentative standards covering textile materials which are referred to earlier in this report. In connection with each title is given the reference to the publication in which the standard appears in its present form.

REVISIONS OF TENTATIVE STANDARDS

Tentative Methods of Testing and Tolerances for Woven Glass Fabrics (D 579 - 40 T):[1]

Section 3.—In the tables in Paragraphs (*a*) and (*b*) change the first two values for nominal thickness from "0.003 in. and under" to read "0.005 in. and under" and "Over 0.003 to 0.010 in." to read "Over 0.005 to 0.010 in."

Tentative Methods of Testing and Tolerances for Woven Glass Tapes (D 580 - 40 T):[2]

Section 3.—Make the same change as recommended above in Section 3 of Methods D 579.

Tentative Methods of Testing Rayon Staple (D 540 - 39 T):[3]

Section 14.—Change the last sentence from its present form to read as follows, including the addition of the note:

> A group of 200 to 700 of the cut fibers shall be weighed to the nearest 0.01 mg. and then the total number of fibers in the group determined by counting. If the group contains less than 200 fibers it shall be discarded and a larger group taken, weighed, and counted.
>
> NOTE.—Counting of the fine fibers is facilitated by transfer across a plush or other short pile surface.

Tentative Methods for Identification of Fibers in Textiles and Quantitative Analysis of Textiles (D 276 - 37 T):[4]

Omit Sections 1 to 9 of these methods comprising the methods for the quantitative analysis of textiles. These sections are being replaced by the new Tentative Methods of Quantitative Analysis of Textiles (D 629 - 41 T), appended hereto.[5]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard General Methods of Testing and Tolerances for Cotton Yarns (D 180 - 40):[6]

Section 2 (b) and (c).—Change from their present form to read as follows:

> (*b*) *Moisture Equilibrium.*—It shall be considered that moisture equilibrium is reached when, after free exposure to air in motion, two successive weighings not less than 15 min. apart show not more than a 1.0 per cent change in weight.
>
> (*c*) *Commercial Regain.*—The commercial regain for cotton yarn shall be 7 per cent of the oven-dry weight.

Standard Methods of Testing and Tolerances for Tubular Sleeving and Braids (D 354 - 36):[7]

Section 11.—Renumber as Section 13 and change from its present form to read as follows:

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[1] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 471.

[2] *Ibid.*, p. 476.

[3] 1939 Book of A.S.T.M. Standards, Part III, p. 1020.

[4] 1939 Book of A.S.T.M. Standards, Part III, p. 951.

[5] These methods were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 537.

[6] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 156.

[7] 1939 Book of A.S.T.M. Standards, Part III, p. 428.

13. *Weight, Yards per Pound.*—(*a*) Determinations of weight shall be made on the samples used for the measurement of inside diameter (Section 11).

(*b*) With the sleeving fitting properly on the specified size of mandrel, 2 ft. in the center section of the sample shall be marked off and cut with a sharp instrument. This portion shall then be weighed to the nearest 0.1 g. and the yards per pound calculated.

Section 13.—Renumber as Section 11 and change from its present form to read as follows:

11. *Inside Diameter.*—(*a*) The inside diameter of sleeving or braid shall be determined by the use of a mandrel 1 yd. in length and of the specified diameter, having a hemispherical end. A sample of sleeving approximately 1¼ yd. in length shall be slipped over the mandrel and, if of the proper size, shall fit smoothly and snugly after application, and show no undue disturbance of the construction due to forcing the sample onto the mandrel.

(*b*) Not less than three determinations shall be made per shipment. Samples shall be taken from different bundles and the average of all determinations shall be considered the average inside diameter of the sleeving.

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Textile Testing Machines (D 76 - 40):[8]

New Sections.—Add new Sections 11 and 12 comprising a description of twist testers to read as follows:

TWIST TESTERS

11. *For Continuous-Filament Single Yarns, and All Plied Yarns.*—(*a*) The twist tester used for determining turns per inch of twist in single and plied yarns of continuous filaments and in plied yarns of staple fibers shall conform to the requirements prescribed in Paragraphs (*b*) to (*e*).

(*b*) The twist tester shall consist essentially of two clamps, one rotatable and the other nonrotatable, suitably mounted opposite and contrary to each other in such a manner that the distance between them is adjustable.

(*c*) The rotatable clamp shall be capable of being revolved in either direction on its axis common to the longitudinal axis of the test specimen and may be hand or power driven. It shall be provided with a counting device indicating the number of revolutions to a minimum accuracy of one turn. The counting device shall be constructed in such a manner that it may be reset readily at the zero mark after each test.

(*d*) The nonrotatable clamp shall be mounted on its support in such a manner that a force may be applied to the clamp to exert a known pull upon the test specimen or no pull as required. A suitable scale graduated in inches and decimal fractions shall be provided to measure the elongation or contraction of the specimen.

(*e*) The distance between the clamps shall be adjustable to accommodate a test specimen from ½ to at least 10 in. in length and the distance shall be indicated on a scale graduated in divisions of ½ in. or less.

12. *For Single Yarns of Staple Fiber.*—(*a*) The twist tester used for determining turns per inch of twist in single yarns of staple fibers shall conform to the requirements prescribed in Paragraph (*b*).

(*b*) The twist tester shall consist of the same elements as prescribed in Section 11 with an additional device for applying tension to the test specimen to employ the untwist-twist method for determining the turns per inch. The device shall be designed so that the tension may be applied to the specimen only at the start and finish of the test and removed completely during the intervening untwisting and twisting operations.

NOTE.—There are two general types of tension devices in which (*1*) the tension is applied manually and controlled by the displacement of a pendulum from its vertical position, and (*2*) the tension is applied manually by the means of a known dead-weight load.

Standard Specifications and Methods of Test for Fineness of Wool Tops (D 472 - 41):[9]

Section 3.—Change to read as follows and add a new Table II (the accompanying Table I), renumbering the subsequent table accordingly:

3. *Fineness.*—The average fiber width and the distribution of single fiber width around the average and other criteria of the fineness of wool top, as determined by test, shall conform to the requirements specified in Tables I and II.

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[8] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 140.

[9] 1941 Supplement to Book of A.S.T.M. Standards, Part III, 179.

TABLE I.—REQUIREMENTS FOR WOOL TOPS.

Grade	48's	46's	44's	40's	36's
Standard values:					
Fineness average[a], microns, $\bar{X}$	32.5	34.0	35.5	37.5	40.0
Standard deviation[a], σ'	7.80	8.20	8.60	9.00	9.60
Limits for test:					
Number of fibers, n	1600	1600	1600	1600	1600
Top limit[a] for v, per cent	25.3	25.3	25.3	25.3	25.3
Grade as indicated if v is less than 25.3 per cent and fineness, $\bar{X}$ is within limits indicated:					
Upper limit	33.12	24.65	36.17	38.21	40.76
Lower limit	31.88	33.35	34.83	36.79	39.24
Heterotypical fibers, max., per cent	1.0	1.0	1.0	1.0	1.0

[a] Where:

$\bar{X}$ = average of 1600 measurements of fiber width or diameter,

σ = standard deviation of 1600 measurements; calculated as in Section 11 and Table III (the present Table II) or as in A.S.T.M. Manual on Presentation of Data.

v = coefficient of variation or the standard deviation of 1600 measurements expressed as a percentage of the average diameter of the fibers.

Standard General Methods of Testing and Tolerances for Cotton Yarns (D 180 – 40):[6]

New Section.—Add under tolerances as a new Section 6 the following requirement for "appearance" of cotton yarns, renumbering the present sections accordingly:

6. *Appearance.*—The appearance of the yarn shall be at least equal to that of the grade specified.

Section 11.—Change Paragraph (*b*) from its present form to read as follows:

(*b*) *Turns in Single Yarn.*—The turns of twist of single yarn shall be determined by the untwist-twist method on any approved type of twist tester. The minimum distance between the clamps shall be 5 in. although a 10-in. distance is preferable. The starting and end tensioning load on the test specimen shall be 3 g. The procedure using the two common methods of tensioning the specimen shall be as follows:

(*1*) *Manually Applied, Tension Pendulum Control.*—The counter of the tester shall be set at the zero mark. The specimen shall be secured in the fixed clamp and placed in the open rotatable clamp. The specimen shall be placed in tension by pulling on it until the pointer attached to the fixed clamp has reached a predetermined position indicating the specified tension on a quadrant in which position the rotatable clamp shall be tightened securely. The rotatable clamp shall be revolved in the direction which untwists the specimen continuing the rotation in the same direction beyond the neutral point thereby imparting a twist to the specimen which is opposite to the original direction. During the first part of the operation the position of the indicator will change as the slack point is approached, but as the clamp is revolved beyond the neutral point, the indicator will return to its initial position where the specimen will be again under the original tension. When this point is reached it is assumed that the same amount of twist has been reinserted as was initially in the specimen. The reading on the counter shall be divided by two and by the distance in inches between the clamps to give the turns per inch.

(*2*) *Manually Applied Tension, Dead-Weight Control.*—The counter of the tester shall be set at the zero mark. The specimen shall be secured in the rotatable clamp and placed in the open fixed clamp while a tensioning load of 3 g. is applied at the center of the specimen in such a manner that a deflection of $\frac{1}{8}$ in. is obtained at the point of application. The fixed clamp shall be tightened securely and the tensioning load removed. The rotatable clamp shall be revolved in the direction which untwists the specimen continuing the rotation in the same direction beyond the neutral point thereby imparting a twist to the specimen opposite to the original direction. When sufficient turns have been inserted to prevent slippage of the fibers the tensioning load shall be reapplied and twisting continued until the deflection of $\frac{1}{8}$ in. is obtained again. When this point is reached it is assumed that the same amount of twist has been reinserted as was initially in the specimen. The reading on the counter shall be divided by two and by the distance in inches between the clamps to give the turns per inch.

NOTE.—It should be recognized that the load of 3 g. for tension is tentative and may be changed. Its tentative use is justified by the preliminary results of an investigation of a variety of yarns which prove it to be nearer the correct value than that obtained by dividing 156 by the yarn number.

Change the last sentence of Paragraph (*c*) to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

The strands shall be gripped in the clamps under a definite tension by applying a load [in grams equal to 156 divided by the yarn number] *of 3 g.*

Omit the note following Paragraph (*c*).

New Section.—Add under methods of testing as a new Section 13 the following test for appearance of cotton yarns:

13. *Appearance:* (*a*) *Grades.*—Photographic standards have been established in Grades *A*, *B*, *C*, and *D*, where *A* is the highest grade and the others are progressively lower. These grades apply to several groups of yarn numbers for each of which there is a separate set of standards. The grouping of yarn numbers is as follows:

3.0 to 7.0
7.0 to 16.5
16.5 to 32.0
32.0 to 65.0
65.0 to 125.0

(*b*) *Test.*—A sample of the yarn under test shall be wound upon a black board of designated size with a designated traverse and compared with a photographic standard (Note).

(*c*) *Number of Tests.*—Five tests shall be made on each case, bale, chain ball, or beam warp. If 80 per cent of the results meet the specified grade and the remaining 20 per cent do not fall below the next lower grade, the yarn shall be considered to be of the grade specified.

NOTE.—A complete set of photographic standards for each group of yarns, together with detailed information regarding the size of board and traverse, may be obtained from the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

The committee recommends that the tentative revision[10] of Section 2 (*c*) be withdrawn in view of the revision proposed for immediate adoption this year,[11] and this definition of standard regain be added as an editorial modification[12] as new Paragraph A3 to the Appendix of Methods D 180.

It is also proposed to include as editorial changes[14] in the Appendix of Methods D 180, the revisions[12] published as tentative for Paragraphs A4 (*c*) and A5. The present new sections on Appearance published as tentative[10] for addition as Sections 6 and 13 are to be continued as tentative.

Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 - 38):[13]

New Sections.—Add new Sections 11 and 12 to read as follows, renumbering the subsequent sections accordingly These two new sections replace the present tentative revision[14] of Section 11, proposed in 1940 and the revision of Section 12, proposed in 1939.

11. *Sampling.*—The sampling of the shipment shall be as representative as possible within practical limitations. A minimum of 25 yd. shall be drawn from the outside of the cone before taking the sample. The sample shall be so handled that there will be no change in twist from the time the sample is drawn until the test is started. In each determination only one observation shall be made on an individual end, whether in the form of woven cord fabric or cord on cones.

12. *Number of Tests.*—The number of tests required for the determination of each physical property of tire cord shall be in accordance with Table I (the accompanying Table II) which also gives the information on which the requirements are based.

TABLE II.—NUMBER OF TESTS.

	Breaking Strength	Yards per Pound	Thickness	Twist
Number of tests, n..	40	10	10	12
Coefficient of variation, V, per cent.	7	2	2	4
Precision, per cent.	2.2	1.5	1.5	2.3
Probability........	0.95	0.95	0.95	0.95

NOTE.—Information available to the committee indicates that the values given in Table I for coefficient of variation, V, are approximately correct, but if the running level of V based on experience is found to be V_1, the number of tests, n_1, required will be determined from the following formula:

$$n_1 = \frac{V_1^2 \times n}{V^2}$$

In no case shall n_1 be less than n.

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[10] 1940 Supplement to Book of A.S.T.M. Standards, Part III, 571.

[11] See p. 449.

[12] See p. 454.

[13] 1939 Book of A.S.T.M. Standards, Part III, p. 431.

[14] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 572.

Standard Methods of Testing Pile Floor Covering (D 418 – 40):[15]

New Section.—Add a new Section 23 to read as follows:

23. *Report.*—A form for reporting the results of tests on pile floor covering is shown in Fig. 1. The use of this form is recommended so as to secure complete and uniform reports throughout the industry.

FIG. 1.—FORM FOR REPORTING RESULTS.[a]

....LABORATORY

PILE FLOOR COVERING ANALYSIS[b]

Manufacturer.. Report No..
Grade name.. Date...
Pattern No.............................. Weave.............................. Type of Back Sizing ....
Pitch per Inch............... Rows per Inch............... Shots............... Frames Working.....................
Dead ....
Sample Dissected (16 sq. in., min.).......................................sq. in. Tuft Length.......................................in.
Thickness Measurements......Total...................in. Back...................in. Pile...............................in.

Weights at Standard Conditions:

	Material	Yarn Number	Ply	Weight as Dissected, oz. per sq. yd.	Clean Weight, oz. per sq. yd.
Pile yarn					
Chain yarn					
Filling yarn					
Stuffer yarn					
			Total		

[a] This form has been approved by A.S.T.M. Committee D-13 on Textile Materials. Analysis shall be performed according to latest edition of Standard Methods of Testing Pile Floor Covering (A.S.T.M. Designation: D 418) of the American Society for Testing Materials, unless otherwise noted.

[b] The results obtained from the analysis of a single sample may vary appreciably from the true average of the grade represented. Reference should be made to Section 17 of the Standard Methods D 418,[15] for estimates of the precision of the above results.

Remarks:

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SIGNED BY..

Standard Definitions of Terms Relating to Textile Materials (D 123 – 40):[16]

Change the definition of Weight, Commercial to read as follows by the addition of the italicized words and the omission of the word in brackets:

> Weight, Commercial.—The oven-dry weight of a textile material *except rayon* plus the weight corresponding to its commercial [moisture] regain.

Add new definitions for commercial weight of rayon and for resilience to read as follows:

> *Weight, Commercial* (*Rayon*).—The oven-dry weight of staple or filament rayon after scouring by definite prescribed methods plus the weight corresponding to its commercial regain.
>
> *Resilience.*—The property of a material by virtue of which it tends to recover its original size and shape after removal of the stress causing deformation; the rate of recovery is without

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[15] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 180.

[16] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 143.

limits and resilience thus includes elasticity (instantaneous recovery).

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

Tentative Methods of Testing and Tolerances for Single Jute Yarn (D 541 – 39 T):[17]

Section 13 (b).—Change the third sentence from its present form: namely,

> The distance between the contact points of the drums or capstans shall be 10 ± ½ in.

to read as follows:

> The distance from center to center of the spools or drums of the testing clamps shall be 10 ± $\frac{1}{16}$ in.

Tentative Definitions of Terms Relating to Textile Materials (D 123 – 40 T):[18]

Change the note accompanying the definition of Moisture Regain, Standard: 1. (Individual Samples) to read as follows by the addition of the italicized figures and the omission of those in brackets:

> NOTE.—The lower moisture regain may be that reached at equilibrium in any atmosphere having a relative humidity between [5] *10* and [50] *40* per cent.

Change the definition of Sliver to read as follows by the addition of the italicized words and the omission of the word in brackets:

> Sliver.—A continuous strand of loosely assembled fibers that is approximately uniform in [diameter] *cross-sectional area* and without twist.

EDITORIAL CHANGES IN STANDARDS

Standard General Methods of Testing and Tolerances for Cotton Yarns (D 180 – 40):[6]

The committee recommends that the following editorial changes be made in the Appendix to Methods D 180:

Add as new Paragraph A3 the revision[10] of Section 2 (*c*) published as tentative since 1938 revised to read as follows renumbering the subsequent sections accordingly:

> A3. *Standard Regain.*—The standard regain for cotton yarn shall be 7.5 per cent of the oven-dry weight.
>
> NOTE.—The 7.5 per cent figure is the average of a number of results obtained on special investigating committee representative yarns which had been brought previously to a standard condition. For any specific yarn, the standard regain may deviate from 7.5 per cent due to variables of origin of the fiber, staple, and twist construction.

Make as editorial changes the revisions in Paragraphs A4 (*c*) and A5 published as tentative[12] since 1938.

Tentative Specifications and Methods of Test for Asbestos Yarns (D 299 – 40 T):[19]

Section 10 (b).—Change the note following this section from its present form to read as follows:

> NOTE.—The factor 0.90 in the formula compensates for an assumed contraction of 10 per cent incurred in twisting.

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[17] 1939 Book of A.S.T.M. Standards, Part III, p. 1003.

[18] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 428.

[19] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 430.

APPENDIX II

PROPOSED METHODS FOR IDENTIFICATION OF FINISHES ON TEXTILES (QUALITATIVE)[1]

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

1. *Scope.*—These methods cover the qualitative procedures for the identification of textile finishes of five types as follows:

Urea-Formaldehyde Resin Finishes,
Straight Formaldehyde Finishes,
Alkyd Resin Finishes Containing Phthalates,
Polymerized Ethylene Derivative Resin Finishes (Polyvinyl or Polyacryl Compounds), and
Nitrocellulose Finishes.

2. *Urea-Formaldehyde Resin Finishes.*—Place a small sample of the fabric to be tested (approximately 3 sq. in.) in a test tube and cover with about 5 ml. of diluted H_2SO_4 (5 per cent) or NaOH (5 per cent). Heat the solution nearly to boiling and allow to stand until cold. Add the supernatant liquid drop by drop to carbazole solution (0.1 per cent by weight in H_2SO_4 (sp. gr. 1.84)) (Note 1). This may best be performed on a porcelain spot plate. If formaldehyde is present as a result of the hydrolysis of the resin, the carbazole solution will be colored a deep blue and a blue precipitate will form on standing (Note 2). The presence of urea-formaldehyde resin in amounts as low as 0.1 per cent of the weight of the fabric may be detected by this test.

Note 1.—It is best to prepare a fresh carbazole solution for each series of tests, or at least daily.

Note 2.—If the fabric is dyed with a color that might obscure the test results, place 10 ml. of the acid or alkaline solution together with the sample in a small distilling apparatus and heat until about 5 ml. of distillate have been collected. Then make the test described in Section 2 on the distillate.

3. *Straight Formaldehyde Finishes.*—This procedure provides a means of distinguishing between a cellulosic fabric which has been rendered crease-resistant or crush-resistant by treatment with formaldehyde alone (treatment A) and a fabric which has been treated with a urea-formaldehyde resin (treatment B).

(*a*) Place a small sample of the fabric to be tested (approximately 3 sq. in.) in a test tube and cover with about 5 ml. of diluted H_2SO_4 (5 per cent). Heat the solution nearly to boiling, allow to stand until cold, and add drop by drop to carbazole solution (see Section 2) on a spot plate. If a blue color appears, either finishing treatment A or B may have been used (Note).

(*b*) Treat a duplicate sample of the fabric with about 5 ml. of NaOH (5 per cent) instead of H_2SO_4 as described in Paragraph (*a*), and add drop by drop to the carbazole solution on a spot plate. The appearance of a blue coloration indicates that treatment B (urea-formaldehyde) has been used. If a blue coloration is not obtained in the second test, but did appear in the first test, it is an indication that treatment A (formaldehyde alone) has been used.

Note.—If dyes which may have been aftertreated with formaldehyde are present, they should be stripped off with sodium hydrosulfite prior to conducting the test.

4. *Alkyd Resin Finishes Containing Phthalates.*—Shred about 0.1 g. of the fabric to be tested and place in a No. 0 porcelain

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[1] These proposed methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials. Published as information, June, 1941.

crucible. Add to this 0.3 g. of resorcinol and 0.5 ml. of H_2SO_4 (sp. gr. 1.84). Place the crucible in an oil bath at 120 ± 2 C. for 10 min. After cooling, take up the residue with 5 ml. of water, transfer with about 10 ml. to a filter, and wash with about 10 ml. more of water. Place the filtrate in a 50-ml. separatory funnel and shake out with 20 ml. of ether. Wash the ether layer with two 10-ml. portions of water, and add 10 ml. of NaOH (10 per cent) to the ether solution. The appearance of a yellowish green fluorescence in the aqueous layer indicates the presence of phthalates.

NOTE.—The presence of phthalate resins in amounts as low as 0.1 per cent of the weight of the fabric may be detected by this test.

5. *Polymerized Ethylene-Derivative Resin Finishes (Polyvinyl or Polyacryl Compounds).*—Immerse the sample to be tested and a white, untreated control sample for 30 min. and at about 70 C. in a dye bath containing 0.1 per cent of Celliton Fast Yellow RRA,[2] remove, and rinse well with water. Then immerse the samples in soap[3] solution (0.1 per cent in distilled water) at 38 to 43 C. until the control sample is practically white. Rinse the test sample to remove the soap solution. If the fabric has been finished with a polyvinyl or polyacryl resin, it will be dyed a deep yellow which is relatively fast to soaping. Untreated fabrics, or those treated with other finishes, will not be stained at all, or with only a faint tint which is largely removed by soaping. The presence of the resins in amounts as low as 1.0 per cent of the weight of the fabric may be detected by this test. The test does not furnish positive identification for amounts as low as 0.1 per cent of the weight of the fabric.

NOTE.—If the fabric to be tested is dyed or printed with colors which might obscure the test, strip off these dyes with sodium hydrosulfite prior to conducting the above test.

6. *Nitrocellulose Finishes.*—Place a small sample of the fabric to be tested on a spot plate and touch with a few drops of diphenylamine solution (0.3 g. of diphenylamine dissolved in 20 ml. of H_2SO_4 (sp. gr. 1.84) and 10 ml. of glacial acetic acid). The development of a deep blue coloration indicates the presence of nitrocellulose.

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[2] See "Alphabetical List of American-Made Dyes," *1940 Year Book*, Am. Assn. of Textile Chemists and Colorists, Vol. XVII, Part IV, p. 373.

[3] The soap used shall conform to the Standard Specifications for Chip Soap (A.S.T.M. Designation: D 496) of the American Society for Testing Materials, 1939 Book of A.S.T.M. Standards, Part III, p. 479.

REPORT OF COMMITTEE D-17

ON

NAVAL STORES

A meeting of Committee D-17 on Naval Stores was held during the annual meeting of the Society in Atlantic City, N. J., on June 26, 1940, at which time the work of the various subcommittees was discussed.

TABLE I.—SUMMARY OF RESULTS OF COLLABORATIVE TESTS ON CRYSTALLIZATION TENDENCY OF ROSIN.

Committee Member	Rosin Sample[a]					
	I	II	III	IV	V	VI
G. A. Hawkins, Southern Pine Chemical Co.	About 25 small nuclei on 25th day. No further growth.	None in 30 days	None in 30 days	None in 30 days	None in 30 days	None in 30 days
J. McE. Sanderson, American Cyanamid Co.	None of samples showed any true crystal formation such as observed in previous test. Samples I, II, and III remained clear for period of 30 days. Samples IV, V, and VI showed many small grains, but no crystals at 24 hr. increasing in number only, but not in size throughout balance of observation period. Samples I, II and III free from crystallizing tendency. Samples IV, V and VI show what might be microscopic crystallization, but which could be termed almost free from visible crystallization tendency.					
W. A. Kirklin, Hercules Powder Co.	50 nuclei after 8 days. About one-tenth of mass crystallized at end of 30 days. Clusters of long needles.	None in 30 days	About 10 nuclei after 15 days. About one-eighth of mass crystallized at end of 30 days. Clusters of long needles.	None in 30 days	None in 30 days	None in 30 days
	Samples II, IV, V, and VI developed slight cloudiness after being stored in oven at 105 C. for short period. Microscopic examination showed this to be small particles of dirt or foreign matter rather than crystals.					
C. F. Speh, U.S. Bureau of Agric. Chemistry and Eng.	Few small nuclei on walls of test tube at 5 days. Growth continued. At 30 days long needles scattered throughout mass.	None in 30 days	Few small nuclei near bottom of tube in 18 days. At 30 days number increased but most of crystals near bottom of test tube.	None in 30 days	None in 30 days	None in 30 days
	Samples observed with aid of polaroid glasses to identify crystals. All samples contained globules of water when placed in oven. This water disappeared after about one week. Sample I contained more crystals than sample III. Crystalline portion of rosin cannot be closely estimated.					

[a] Identification of samples used in crystallization test:
I. Wood rosin from Newport Co.,
II. Gum rosin as loaded in tank car ready for shipment,
III. Gum rosin II on arrival at destination 5 days later,
IV. Gum rosin made on steam still,
V. Gum rosin made on steam still, and
VI. Gum rosin made on fire still.

None of the subcommittees has undertaken during the year any new collaborative tests.

Subcommittee I on Softening Point of Rosin (C. E. Kinney, chairman) has conferred with the Special Committee on Softening Point Test, of Committee E-1 on Methods of Testing, for the purpose of developing a shouldered ring for use in the Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (E 28 – 39 T) which will be suitable for all classes of products. The ring was first modified to make the inside bottom diameter correspond to that of the tapered ring and to slightly increase the top outside diameter. The second modification further increased the outside top diameter in order to give greater strength. A number of these rings were made and submitted to various members of Committee D-17 and the special committee of Committee E-1 for comment.

The collaborative tests on six samples of rosin by Subcommittee II on Crystallization (E. V. Romaine, chairman) were completed. A summary of results is submitted as shown in Table I.

Subcommittee IV on Acid and Saponification Number and Unsaponifiable Matter (C. C. Zeigler, chairman) has held further collaborative work in abeyance pending results of tests on titration conducted in Mr. Zeigler's laboratory involving the use of inside indicators outside indicators, two-phase solution with inside indicator, and electrometric titration.

This report has been submitted to the letter ballot of the committee, which consists of 20 members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. P. VEITCH,
Chairman.

C. F. SPEH,
Secretary.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES

Committee D-18 on Soils for Engineering Purposes has held three meetings during the past year: at Atlantic City, N. J. on June 26, 1940; in Washington, D. C., on December 4, 1940; and in Washington, D. C., on March 5, 1941.

Plans are being made by the committee to sponsor a Symposium on Tests of Soils for Engineering Purposes, to be presented at the 1942 annual meeting of the Society. The eleven subcommittees will participate, and will sponsor papers concerning the present status of tests as it relates to the methods being considered by them. Special attention will be paid to descriptions of the different test methods now being used.

The committee has no recommendations affecting standards to present to the Society at this annual meeting. The Method of Test for Water Absorption and Resistance to Plastic Flow of Soil and Emulsified Asphalt Mixtures was recommended for publication as tentative at the 1940 annual meeting, but action was taken to refer this recommendation to Committee E-10 on Standards. No further action with respect to this test is being recommended by the committee at this time.

Several subcommittees are studying tests, with the expectation that reports on them will be ready for submission to the Society at the 1942 annual meeting.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (W. P. Kimball, chairman) has prepared the Proposed Definitions of Terms and Symbols Relating to Soil Mechanics, appended hereto.[1] Copies have been distributed to the membership of Committee D-18 for review and criticism. These proposed definitions and symbols are being published in this report as information. It is planned to submit them to letter ballot of the committee following the annual meeting of the Society, with the expectation that they may be presented to the Society subsequent to the June meeting through Committee E-10 on Standards.

Subcommittee IV on Methods of Testing the Physical Characteristics of Soils (Paul Rapp, chairman) has submitted for consideration of the committee a new Proposed Method of Test for Compaction and Density of Soil, which is being published as information as appended hereto.[2] It is planned also to submit this method to letter ballot of the committee. It has also been referred to Committee D-4 on Road and Paving Materials for consideration.

This report has been submitted to letter ballot of the committee, which consists of 75 members; 47 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. A. HOGENTOGLER,
Chairman.

WESLEY R. NELSON,
Secretary.

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[1] See p. 460.
[2] See p. 467.

APPENDIX I

PROPOSED DEFINITIONS OF TERMS AND SYMBOLS RELATING TO SOIL MECHANICS[1]

These are proposed definitions and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

PART I.—SYMBOLS AND TERMS RELATING TO SOIL MECHANICS.

Symbol	Term
A	Area (**II**)[2]
a_v	Compressibility, coefficient of (**V**)
b	Breadth, or width (**II**)
C	Cohesion, resultant or total (**IV, E**)
C_c	Compression index (**V**)
C_s	Swelling index (**V**)
C_u	Uniformity coefficient (Hazen) (**III**)
c	Cohesion per unit area (**IV, E**)
c_v	Consolidation, coefficient of (**V**)
D	Diameter, or grain diameter, (**II, III**)
D_{10}	Effective size (Hazen) (**III**)
D_d	Density, degree of, or compaction, degree of (**III**)
E	Modulus of elasticity (**IV, D**)
e	Base of natural logarithms, or void ratio (**II, III**)
e_c	Void ratio, critical (**III**)
e_o	Void ratio at pressure p_o on the compression curve (**V**)
e_f	Flocculation ratio (**III**)
e_s	Void ratio at pressure p_o on the swelling curve (**V**)
F	Force, total internal (**IV, A**)
F_n	Normal component of F (**IV, A**)
F_t	Tangential (shearing) component of F (**IV, A**)
f	Force per unit area, or stress per unit area, internal (**IV, A**)
f_n	Normal component of f (**IV, A**)
f_t	Tangential (shearing) component of f (**IV, A**)
$\bar{F}$	Effective force, total internal (**IV, A**)
$\bar{F}_n$	Normal component of $\bar{F}$ (**IV, A**)
$\bar{F}_t$	Tangential (shearing) component of $\bar{F}$ (**IV, A**)
$\bar{f}$	Effective force per unit area, or effective stress per unit area, internal (**IV, A**)
$\bar{f}_n$	Normal component of $\bar{f}$ (**IV, A**)
$\bar{f}_t$	Tangential (shearing) component of $\bar{f}$ (**IV, A**)
G	Specific gravity (**III**)
g	Acceleration of gravity (**II**)
H	Depth, height, thickness (**II**)
H_s	Reduced height (**II**)
I	Modulus of incompressibility (**IV, D**)
I_c	Consistency index (**III**)
I_f	Flow index (**III**)
I_p	Plasticity index (**III**)
I_R	Remolding index (**III**)
I_T	Toughness index (**III**)
I_w	Moisture index (**III**)
i	Hydraulic gradient (**II**)
J	Seepage force, resultant (**II**)
j	Seepage force per unit volume (**II**)
K	Hydrostatic pressure ratio (**III**)
k	Permeability, coefficient of (**III**)
L	Distance, or length (**II**)
L_E	Lineal expansion (**III**)
L_{LE}	Lineal expansion limit (**III**)
L_{LS}	Lineal shrinkage limit (**III**)
L_S	Lineal shrinkage (**III**)
m	Perimeter shear (**IV, E**)
n	Porosity, or developed pressure (**II; III; IV, E**)
P	Force, total, or load, total (**IV, B**)
P_A	Earth pressure, total active (**IV, B**)

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[1] These proposed definitions and symbols are under the jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes. Published as information, June, 1941.

[2] The boldface roman numerals in parentheses indicate that a further definition of the symbol and term appears in a later part, that is, in Parts II to V, which cover separate classifications of the terms as to properties.

P_P	Earth pressure, total passive **(IV, B)**
p	Force per unit area, or load per unit area **(IV, B)**
Q	Discharge, rate of, or resistance, total or resultant **(II; IV, E)**
q	Discharge, rate of per unit length or per unit area, or resistance per unit area or length **(II; IV, E)**
q_o	Ultimate bearing capacity per unit area at ground surface **(IV)**
q_r	Compressive strength per unit area (triaxial) **(IV, E)**
q_s	Shear strength per unit area **(IV, E)**
q_u	Compressive strength per unit area (unconfined test)**(IV, E)**
R	Reading **(II)**
R_s	Shrinkage ratio **(III)**
r	Radius **(II)**
S	Hydraulic gradient; or saturation, degree of **(III)**
s	Shear strength per unit area **(II; IV, E)**
T	Temperature **(II)**
T_v	Time factor **(V)**
t	Time **(II)**
U	Neutral force **(V)**
$U\%$	Consolidation, percentage of **(V)**
u	Hydrostatic excess pressure **(V)**
u_w	Neutral pressure per unit area **(V)**
u_z	Hydrostatic pressure **(V)**
V	Volume **(II)**
V_S	Volumetric shrinkage **(III)**
V_{SL}	Volumetric shrinkage limit **(II)**
v	Velocity **(II)**
W	Weight, or load, total **(II; IV, B)**
$W\%$	Percentage finer matter in mechanical analysis plot **(III)**
w	Water content (moisture content) **(III)**
w_{cme}	Centrifuge moisture equivalent **(III)**
w_f	Flocculation limit **(III)**
w_{fme}	Field moisture equivalent **(II)**
w_l	Liquid limit **(III)**
w_o	Water content, optimum **(III)**
w_p	Plastic limit **(III)**
w_s	Shrinkage limit **(III)**
α	Angle **(II)**
β	Angle **(II)**
γ	Unit weight **(III)**
γ_w	Unit weight of water, equal to 1 in the metric system
δ	Displacement, or deformation, total (see also ρ) **(IV, C)**
δ_x δ_y δ_z	Components of δ **(IV, C)**
ϵ	Base of natural logarithms, or deformation per unit length, or strain **(II; IV, C)**
ζ	Component of ρ in Z direction **(IV, C)**
η	Component of ρ in y direction **(II; IV, C)**
θ	Angle **(II)**
μ	Coefficient of absolute viscosity
ν	Coefficient of kinematic viscosity, or concentration factor **(II; IV, A)**
ξ	Component of ρ in x direction **(IV, C)**
ρ	Displacement, or deformation, total (see also δ) **(IV, C)**
ρ_x ρ_y ρ_z	Components of ρ **(IV, C)**
σ	Normal component of internal force per unit area (normal component of stress) **(IV, A)**
$\bar{\sigma}$	Normal component of effective internal force per unit area (normal component of stress) **(IV, A)**
τ	Tangential component of internal force per unit area (tangential component of stress) **(IV, A)**
$\bar{\tau}$	Tangential component of effective internal force per unit area (tangential component of effective stress) **(IV, A)**
ϕ	Angle of internal friction **(III)**
ψ	Angle **(II)**

Greek Alphabet

Α α	alpha	Η η	eta	Ν ν	nu	Τ τ	tau
Β β	beta	Θ θ ϑ	theta	Ξ ξ	xi	Υ υ	upsilon
Γ γ	gamma	Ι ι	iota	Ο ο	omicron	Φ ϕ φ	phi
Δ δ	delta	Κ κ	kappa	Π π	pi	Χ χ	chi
Ε ε	epsilon	Λ λ	lambda	Ρ ρ	rho	Ψ ψ	psi
Ζ ζ	zeta	Μ μ	mu	Σ σ ς	sigma	Ω ω	omega

PART II.—GENERAL TERMS.

Acceleration of Gravity, g.

Angle, α, β, θ, ψ.—Angles are generally best designated by Greek letters. These four letters have no specific application in this list, and are therefore convenient for designating angles.

Area, A.

Base of Natural Logarithms, e or ϵ.—The symbol, e, is preferred, but where its use might cause confusion with its use designating void ratio the symbol, ϵ, should be used.

Breadth, b.

Coefficient of Absolute Viscosity, μ.

Coefficient of Dynamic Viscosity, ν.—Defined by the equation $\nu = \dfrac{\mu}{\gamma_f}$, where γ_f = the unit weight of the fluid in question.

Depth, H.

Diameter, D.

Discharge, Rate of, Q.—In soil mechanics literature this usually refers to the rate at which water is discharged through a given area of soil. Dimensionally, Q = volume/time. In Darcy's familiar equation for laminar flow $Q = kSA$.

Discharge, Rate of per Unit Length or per Unit Area, q.—Dimensionally, q = volume/(time × area), or q = volume/(time × length). In Darcy's equation as used above $q = kS$.

Distance, L.

Height, H.

Hydraulic Gradient, S or i.—The symbol, S, is preferred because of its widespread use in literature on hydraulics. The symbol, i, is included because it has been almost exclusively used to designate hydraulic gradient in soil mechanics literature.

Length, L.

Radius, r.

Reading, R.—For example, reading of a deflection dial, hydrometer, and similar apparatus.

Reduced Height, H_s.—The volume of the solid particles in a given soil mass divided by the cross-sectional area of the mass. Usually applied to a soil stratum where it represents the thickness or height which the stratum would have if its void ratio were zero. Expressed by the equation $H_s = \dfrac{H}{(1+e)}$. Note that in consolidation calculations the height and reduced height of a stratum draining at both faces are generally designated by $2H$ and $2H_s$, respectively.

Seepage Force, Resultant, J.

Seepage Force per Unit Volume, j.—Note that $j = S\gamma_f$, where γ_f is the unit weight of the fluid.

Temperature, T.

Thickness, H.

Time, t.

Velocity, v.

Volume, V.

Weight, W.

Width, b.

PART III.—TERMS USED IN THE DESCRIPTION OF SOILS AND THEIR PROPERTIES.

Angle of Internal Friction, ϕ.

Centrifuge Moisture Equivalent, w_{cme}.—In most uses the abbreviation *CME* may be used. When, as rarely, a symbol is desired, w_{cme} should be used. The *CME* is the water content retained by a soil which has been first saturated with water and then subjected to a force equal to one thousand times the force of gravity for one hour.

Compaction, Degree of, D_d.—Also called degree of density. Defined by the equation:

$$D_d = \frac{\text{(Void ratio of sample)} - \text{(Void ratio in densest state)}}{\text{(Void ratio in loosest state)} - \text{(Void ratio in densest state)}}$$

Consistency Index, I_c.—The ratio of the difference between the liquid limit and the natural water content to the difference between the liquid limit and the plastic limit. Defined by the equation:

$$I_c = \frac{w_L - w}{I_p}$$

Density, Degree of.—See Compaction, Degree of.

Diameter, Grain, D.—Also called grain size, or particle size. The size of grain, usually in millimeters, as determined by sieve analysis or wet mechanical analysis; hence not a true grain size except for spherical grains.

Effective Size (Hazen), D_{10}.—The grain size on a mechanical analysis curve corresponding to $W\% = 10$.

Equivalent Volumetric Change.—See Volumetric Shrinkage Limit.

Field Moisture Equivalent, w_{fme}.—In most uses the abbreviation *FME* may be used. When, as rarely, a symbol is desired w_{fme} should be used. The *FME* is the minimum water content at which a drop of water placed on a smoothed surface of a soil will not immediately be absorbed by the soil but will spread over the surface and give it a shiny appearance.

Flocculation Limit, w_f.—The water content of a soil when it is in the condition of a deflocculated sediment.

Flocculation Ration, e_f.—The void ratio of a soil when it is in the condition of a deflocculated sediment.

Flow Index, I_f.—The slope of the flow curve. The flow curve is the locus of points obtained from a standard liquid limit test and plotted on a graph representing water content as ordinates on an arithmetic scale and number of blows as abscissas on a logarithmic scale. Thus the flow index is numerically equal to the difference between the water content at 10 and at 100 blows, or at 1 and at 10 blows.

Hydrostatic Pressure Ratio, K.—A term used in earth pressure computations to represent the ratio between the normal component of pressure at a point on a given plane and the pressure which would exist at the same point in a liquid of the same unit weight. Thus: $p_n = K\gamma_m H$.

Lineal Expansion, L_E.—The increase in one dimension, expressed as a percentage of that dimension at the shrinkage limit, of a soil mass when the water content is increased from the shrinkage limit to any given water content.

Lineal Expansion Limit, L_{LE}.—The increase in one dimension, expressed as a percentage of that dimension at the shrinkage limit, of a soil mass when the water content is increased from the shrinkage limit to the field moisture equivalent.

Lineal Shrinkage, L_S.—The decrease in one dimension, expressed as a percentage of that dimension originally, of a soil mass when the water content is reduced from the original percentage to the shrinkage limit.

Lineal Shrinkage Limit, L_{LS}.—The decrease in one dimension, expressed as a percentage of that dimension at the field moisture equivalent, of a soil mass when the water content is reduced from the field moisture equivalent to the shrinkage limit.

Liquid Limit, w_L.—In most cases the abbreviation *LL* may be used. When, as rarely, a symbol is desired, w_L should be used. The *LL* is the upper limit of the plastic state, expressed as the water content at which the flow curve (see Flow Index) intersects the "25 blows" ordinate.

Moisture Content.—See Water Content.

Moisture Index, I_w.—The ratio of the plasticity index to the difference between the natural water content and the plastic limit expressed by the equation:

$$I_w = \frac{I_p}{w - w_p}$$

Percentage Finer, $W\%$.—The ordinate of the mechanical analysis curve, representing, for a given soil, the percentage of particles by weight having diameters smaller than the value of D corresponding to $W\%$.

Permeability, Coefficient of, k.—The discharge velocity of flow of a fluid through a porous mass under a unit hydraulic gradient. Thus in Darcy's equation, $V = kSAt$, for laminar flow, $k = \frac{V}{At}$, where A is the gross area of the soil mass.

Plasticity Index, I_p.—In most cases the abbreviation *PI* may be used. When, as rarely, a symbol is desired I_p should be used. The *PI* is the numerical difference between the water content at the liquid limit and the water content at the plastic limit. $I_p = w_L - w_p$.

Plastic Limit, w_p.—In most cases the abbreviation *PL* may be used. When, as rarely, a symbol is desired, w_p should be used. The *PL* is the lower limit of the plastic state, expressed as the minimum water content at which a soil can be rolled into a thread $\frac{1}{8}$ in. in diameter without crumbling.

Porosity, n.—The ratio, expressed as a percentage, of the intergranular space in a given soil mass to the total volume of the soil mass. Porosity must be clearly differentiated from void ratio, e. The relation between the two terms is expressed by the equation: $n = \frac{e}{(1+e)}$.

Remolding Index, I_R.—The ratio of the modulus of elasticity (or deformation) of a soil in the undisturbed state to the modulus of elasticity (or deformation) of the soil in the remolded state.

Saturation, Degree of, S.—The ratio, expressed as a percentage, of the volume of water in a given soil mass to the total volume of intergranular space (voids). Thus: $S = \frac{V_w}{V_v} \times 100$.

Shrinkage Limit, w_s.—In most cases the abbreviation *SL* may be used. When, as rarely, a symbol is desired, w_s should be used. The *SL* is the maximum water content at which a reduction in water content will not cause a decrease in volume of the soil mass.

Shrinkage Ratio, R_s.—In most cases the abbreviation *SR* may be used. When, as rarely, a symbol is desired, R_s should be used. The *SR* is the ratio between a given volume change, expressed as a percentage of the dry volume, and the corresponding change in water content above the shrinkage limit, expressed as a percentage of the weight of the oven-dried soil.

Specific Gravity, G.—The ratio of the weight in air of a given volume of a material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature, usually 4 C.

Toughness Index, I_T.—In most cases the abbreviation *TI* may be used. When, as rarely, a symbol is desired, I_T should be used. The *TI* is the ratio of the plasticity index to the flow index. Thus $I_T = \frac{I_p}{I_f}$.

Uniformity Coefficient (Hazen), C_u.—The ratio of D_{60} to D_{10} as determined in a mechanical analysis.

Unit Weight, γ.—Weight per unit volume, as grams per cubic centimeters, pounds per cubic foot, etc.

Void Ratio, e.—The ratio of the volume of intergranular space to the volume of solid particles in a given soil mass without regard to the proportions of liquid, air, or gas which may occupy the space.

Void Ratio, Critical, e_c.—The void ratio of a cohesionless soil at which continuous shearing deformation can take place without change in void ratio.

Volumetric Change.—See Volumetric Shrinkage.

Volumetric Shrinkage, V_S.—Also called volumetric change. The decrease in volume, expressed as a percentage of the dry volume, of a soil mass when the water content is reduced from a given percentage to the shrinkage limit.

Volumetric Shrinkage Limit, V_{SL}.—The decrease in volume, expressed as a percentage of the dry volume, of a soil mass when the water content is reduced from the field moisture equivalent to the shrinkage limit.

Water Content, w.—The ratio, expressed as a percentage, of the weight of water in a given soil mass to the weight of solid particles.

Water Content, Optimum, w_o.—The water content at which the maximum density is produced in a soil by a specific amount of compaction.

PART IV.—TERMS RELATING TO STRESS, STRAIN, AND DISPLACEMENT.

Section A: Internal Stresses and Forces

Concentration Factor, ν.—Factor used in Frölich's and Griffith's theory of stress distribution, as in the equation:

$$f_z = \frac{\nu P}{2\pi R^2}\left(\frac{z}{R}\right)^\nu$$

Force, Total Internal, F.—The total force acting on a given area within a body or within a mass of earth.

Force per Unit Area, or Stress per Unit Area, Internal, f.—The force per unit area or stress per unit area acting at a point or on a surface within a body or within a mass of earth.

Effective Force, Total Internal, $\bar{F}$.—The total force acting on the soil structure over a given area within a body or within a mass of earth, for example, $\bar{F} = F - U$.

Effective Force per Unit Area, or Effective Stress per Unit Area, Internal, $\bar{f}$.—The force per unit area or stress per unit area acting on the soil structure at a point or on a surface within a body or within a mass of earth, for example, $\bar{f} = f - u$.

Normal Component of These Forces and Stresses, F_n, $\bar{F}_n$, f_n, or σ; $\bar{f}_n$ or $\bar{\sigma}$.

Tangential (or Shearing) Component of These Forces and Stresses, F_t, $\bar{F}_t$, f_t or τ; $\bar{f}_t$ or $\bar{\tau}$.

Section B: External Forces, Pressures, and Loads

Earth Pressure, Total Active, P_A.—The total force exerted by or through a mass of earth against a medium of lateral support or against a given area thereof.

Earth Pressure, Total Passive, P_P.—The total externally applied lateral force which a given mass of earth is capable of resisting over a given area.

Force, Total, P.—Externally applied force.

Force per Unit Area, p.—Intensity of externally applied force.

Load, Total, P or W.—Externally applied load.

Load per Unit Area, p.—Intensity of externally applied load.

Section C: Strains and Displacements

Displacement, δ or ρ.—Translation of a body or mass through space.

Deformation, Total, δ or ρ.—Total deformation of a body or mass within itself, usually caused by externally applied forces.

Components of Displacement or Total Deformation.—The following are used to to identify components of displacement or total deformation:

Components in the x-Direction, δ_x or ρ_x or ξ.

Components in the y-Direction, δ_y or ρ_y or η.

Components in the z-Direction, δ_z or ρ_z or ζ.

Deformation per Unit Length, ϵ.

Strain, ϵ.

Section D: Moduli

Modulus of Elasticity, E.—The ratio of stress to strain for a given material under given load conditions. Subscripts may be used to differentiate moduli under different load conditions.

Modulus of Incompressibility, I.—The ratio of the developed pressure to the volume change in a soil mass of unit volume.

Section E.: Resistances and Strengths

Cohesion per Unit Area, c.—The shearing strength per unit area under zero externally applied load, as used in Rankine's equation: $s = c + p \tan \phi$. The value of c is not a constant for a given soil, but varies according to the test procedure.

Cohesion, Resultant or Total, C.—The total cohesion on a given area, that is, $C = cA$.

Developed Pressure, n.—The stress reaction of a soil mass acting as a pressure per unit area independent of the dimension of the loaded area, and consisting of pressure, components developed by the mass due to resistance to displacement.

Perimeter Shear, m.—The boundary stress reaction per unit length of perimeter developed by a loaded area, a measure of that proportion of the applied pressure which is carried by the body of soil as a function of the perimeter of the bearing area.

Resistance, Total Resultant, Q.—A general symbol to be used with appropriate subscripts where necessary.

Resistance per Unit Area or Length, q.—A general symbol to be used with appropriate subscripts where necessary.

Compressive Strength per Unit Area, q_u.—Compressive strength as determined by an unconfined compression test.

Compressive Strength per Unit Area, q_r.—Compressive strength as determined by a triaxial compression test. Note that $q_r = \sigma\text{I} - \sigma\text{III}$ at failure.

Shear Strength per Unit Area, q_s or s.

Ultimate Bearing Capacity per Unit Area at Ground Surface, q_o.—In an homogeneous soil, the ultimate bearing capacity increases with depth. Ultimate bearing capacities at various depths may be designated by q with suitable subscripts.

PART V.—TERMS RELATING TO THE CONSOLIDATION OF SOILS.

Compressibility, Coefficient of, a_v.—Numerically equal to the slope of the pressure-void ratio diagram obtained from a consolidation test, expressed by the equation: $a_v = -\frac{de}{dp}$.

Compression Index, C_c.—Compression index for a clay sample in the undisturbed state expressed by the equation: $e = e_o - C_c \log \frac{p}{p_o}$. Use C_c' for a clay sample in the remolded state.

Swelling Index, C_s.—Swelling index for a clay sample in the undisturbed state, expressed by the equation: $e = e_s - C_s \log \frac{p}{p_o}$. Use C_s' for a clay sample in the remolded state.

Void ratio at pressure p_o (usually 1 kg. per sq.cm.) on the compression curve obtained by a consolidation test on a clay sample in the undisturbed state, e_o. Use e_o' for a clay sample in the remolded state.

Void ratio at pressure p_o (usually 1 kg. per sq.cm.) on the swelling curve obtained by a consolidation test on a clay sample in the undisturbed state, e_s. Use e_s' for a clay sample in the remolded state.

Consolidation, Coefficient of, c_v.—Defined by Terzaghi by the equation:

$$c_v = \frac{k(1+e)}{a_v \gamma_w}$$

NOTE.—Readers of literature published prior to 1935 should observe that the coefficient of consolidation, usually designated by c, was then defined by the equation $c = \frac{k}{a_v \gamma_w (1+e)}$. This original definition of coefficient of consolidation may be found in some more recent papers and care should be taken to avoid confusion.

Consolidation, Percentage of, $U\%$.—Average degree of theoretical consolidation at a given time within a clay stratum, expressed as a percentage of the total consolidation obtainable under the given stress condition.

Time Factor, T_v.—A dimensionless factor used in the calculation of the rate of consolidation. The value of T_v corresponding to a given percentage consolidation, $U\%$, depends solely on the distribution of stress increment within the consolidating stratum.

Hydrostatic Pressure, u_z.

Neutral Pressure per Unit Area, u_w.—The total pressure per unit area of soil in the pore water at a given time.

Neutral Force, U.—The total pressure, over a given area of soil, in the pore water at a given time expressed by the equation: $U = u_w A$.

Hydrostatic Excess Pressure, u.—The pressure per unit area of soil which exists in the pore water at any time in excess of the hydrostatic pressure, expressed by equation: $u = u_w - u_z$. Note also that $u = f - \bar{f}$.

APPENDIX II

PROPOSED METHOD OF TEST FOR MOISTURE-DENSITY RELATIONS OF SOIL[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 260 S. Broad St., Philadelphia, Pa.

Scope

1. This method of test is intended for determining the relationship between the moisture content of soils and resulting densities (oven-dry weight per cubic foot) when the soil is compacted in the laboratory as specified herein.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Mold.*—A cylindrical metal mold having a capacity of $\frac{1}{30}$ cu. ft. with an internal diameter of 4.0 in. and a height of approximately 4.6 in., which has a detachable collar assembly approximately $2\frac{1}{2}$ in. in height, to permit preparing compacted specimens of soil-water mixtures 4.0 in. in diameter and approximately 4.6 in. in height which have a volume of $\frac{1}{30}$ cu. ft. The mold and collar shall be fastened to a detachable base. (See Fig. 1.)

(*b*) *Rammer.*—A metal rammer having a 2-in. diameter circular face and weighing 5.5 lb. The rammer shall be equipped with a suitable arrangement to control the specified drop.

(*c*) *Sleeve.*—A closed cylindrical sleeve slightly less than 4.0 in. in diameter, or similar device, for removing compacted specimens from the mold.

(*d*) *Balances.*—A balance or scale of 25-lb. capacity sensitive to 0.01 lb., and a 100-g. capacity balance sensitive to 0.1 g.

(*e*) *Drying Oven.*—A thermostatically controlled drying oven capable of maintaining temperatures of about 110 C. (230 F.) for drying moisture samples.

(*f*) *Straightedge.*—A steel straightedge 12 in. in length.

Sample

3. A 6-lb. sample of soil taken from a portion of the material passing the No. 4 (4760-micron) sieve shall be air-dried to a slightly damp condition.

Procedure

4. (*a*) The 6-lb. sample shall be thoroughly mixed, then compacted in the cylinder (with the extension attached) in three equal layers, each layer receiving 25 blows from the rammer dropping free from a height of 12 in. above the elevation of each finally compacted layer. During compaction, the mold shall rest on a uniform, rigid foundation weighing 200 lb. or of equivalent rigidity. The blows shall be uniformly distributed over the surface of the layer being compacted. The extension shall then be removed and the compacted soil carefully trimmed even with the top of the cylinder by means of the straightedge, and weighed.

(*b*) The weight of the compacted sample and cylinder, minus the weight of the cylinder, shall then be multiplied by 30 and the result recorded as the wet weight per cubic foot of the compacted soil.

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[1] This proposed method is under the joint jurisdiction of the A.S.T.M. Committee D-18 on Soils for Engineering Purposes and Committee D-4 on Road and Paving Materials. Published as information June, 1941.

(*c*) The material shall be removed from the cylinder and sliced vertically through the center. At least a 100-g. sample shall be taken from the center, weighed immediately, and dried in an oven at 110 C. (230 F.) for at least 12 hr. or to constant weight to determine the moisture content.

(*d*) The remainder of the material shall be broken up until it will pass a No. 4 (4760-micron) sieve. Water in sufficient amounts to increase the moisture content of the soil sample by approximately 1 per cent shall be added and the above procedure repeated for each increment of water added. This series of determinations shall be continued until the soil becomes very wet or there is a substantial decrease in the wet weight of the compacted soil.

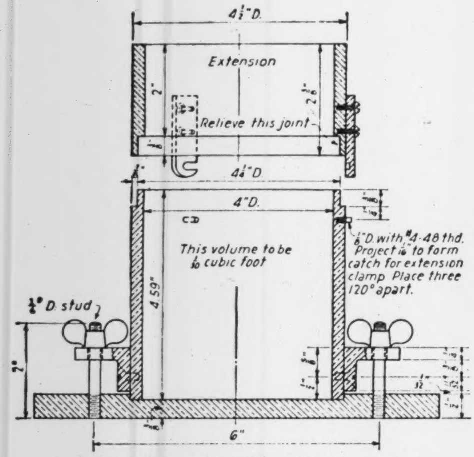
4½" D.
Extension
2"
2⅛"
Relieve this joint
4¼" D
4" D.
This volume to be 1/30 cubic foot
4.59"
⅛" D. with #4-48 thd. Project 1/16" to form catch for extension clamp Place three 120° apart.
⅜" D. stud
2"
6"

FIG. 1.—Cylindrical Mold for Soil Tests.

Calculations

5. The moisture content and the dry weight of the soil as compacted shall be calculated as follows:

$$M = \frac{A - B}{B - C} \times 100$$

$$W = \frac{W_1}{M + 100} \times 100$$

where:

M = percentage of moisture in the specimen,
A = weight of dish and wet soil,
B = weight of dish and dried soil,
C = weight of dish,
W = dry weight per cubic foot of compacted soil, and
W_1 = wet weight in pounds per cubic foot.

Moisture-Density Relationship

6. (*a*) The calculations in Section 5 shall be made to determine the moisture content and corresponding compacted oven-dry weight (density) of the compacted soil for each test made. The oven-dry weight per cubic foot (density) of the soil shall be plotted as ordinates and corresponding moisture contents as abscissas.

(*b*) *Optimum Moisture Content.*—When the moisture-density relations have been determined for a soil and the results plotted as indicated in Paragraph (*a*), it will be found that by connecting the plotted points with a smooth line a curve is produced which is, generally, parabolic in form. The moisture content producing the peak of the curve shall be termed the "optimum moisture content" of the soil under the above compaction.

(*c*) *Maximum Density.*—The oven-dry weight per cubic foot of the soil at "optimum moisture content" shall be termed "maximum density" under the above compaction.

REPORT OF COMMITTEE D-19
ON
WATER FOR INDUSTRIAL USES

Committee D-19 on Water for Industrial Uses and its subcommittees held meetings at Atlantic City, N. J., on June 24 and 25, 1940. No additional meetings were held by any of the subcommittees during the year.

The committee sponsored a session on water at the 1940 annual meeting, in the form of a Symposium on the Problems in the Classification of Natural Water Intended for Industrial Use in which four papers[1] were presented and discussed.

The committee is sponsoring a water session at this annual meeting of the Society in the form of a Symposium on Problems and Practices in Determining Steam Purity by Conductivity Methods. This symposium of very timely interest has been arranged by R. E. Hall, the secretary of the committee, who will serve as chairman of the session and will also present an introduction to the symposium. The papers[2] comprising the symposium are as follows:

"The Sampling of Steam and Boiler Water," by A. R. Belyea and A. H. Moody, Consolidated Edison Company of New York, Inc.

"Experimental Methods of Determining Conductivity Correction Factors for Dissolved Gases in Steam Condensate," by S. F. Whirl and W. A. Lower, Duquesne Light Co.

"Calculation of Corrections to Conductivity Measurements for Dissolved Gases," by D. S. McKinney, Carnegie Institute of Technology.

"The Degasification of Steam Samples for Conductivity Tests," by P. B. Place, Combustion Engineering Co., Inc.

"A New Type of Conductivity Apparatus for Use with Boiler Waters and Steam Samples," by A. R. Mumford, Consolidated Edison Company of New York, Inc.

"Conductivity Cells and Electrical Measuring Instruments," by C. E. Kaufman, Hall Laboratories, Inc.

It is believed that these papers and the discussion will be of value to the committee and its subcommittees in making available data that will permit the development of methods of testing and sampling.

The Society was represented by R. E. Hall, substituting for the official representative, at a meeting of the Subcommittee on Care of Steam Boilers in Service of the Boiler Code Committee of the American Society of Mechanical Engineers. A section has been organized in this subcommittee to undertake the work of suggesting revisions in the paragraphs on water chemistry that now appear in the A.S.M.E. Boiler Construction Code, 1940 Edition, particularly in Section VII on Suggested Rules for Care of Power Boilers. The proposed revisions should merit the attention and cooperation of interested members of Committee D-19, as well as members of the Society who are responsible for boiler operations.

ADOPTION OF TENTATIVE STANDARDS AS STANDARD

The committee recommends that the following tentative methods be approved, without revision, for reference to letter ballot of the Society for adoption as standard:

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 1305, 1307, 1317, 1329 and 1342 (1940).

[2] See pp. 1261 to 1340.

Tentative Methods of:

- Sampling Plant or Confined Waters for Industrial Uses (D 510 – 40 T),[3]
- Reporting Results of Analysis of Industrial Waters (D 596 – 40 T),[3] and
- Determination of Hydroxide Ion in Industrial Waters (D 514 – 38 T).[4]

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 62 members; 45 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
ADOPTION OF TENTATIVE STANDARDS AS STANDARD			
Sampling Plant and Confined Waters for Industrial Uses (D 510 – 40 T)	38	1	6
Reporting Results of Analysis of Industrial Waters (D 596 – 40 T)	37	2	6
Determination of Hydroxide Ion in Industrial Waters (D 514 – 38 T)	36	0	9

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II, Editorial (E. P. Partridge, chairman).—The principal activity of this subcommittee has been its editorial assistance to the other subcommittees. Subsequent to the 1940 annual meeting the chairman cooperated in editing the revisions to the methods discussed at the time of the meeting.

Subcommittee III on Methods of Sampling (M. A. Krimmel, chairman).—This subcommittee has voted in favor of adopting as standard this year, without further revision, the Tentative Methods of Sampling Plant or Confined Waters for Industrial Uses (D 510 – 40 T). The plan of the subcommittee is to develop methods in accordance with the outline previously developed. However, because of pressure of company duties, the chairman of the subcommittee had to defer any committee plans. Mr. D. K. French has accepted the appointment as acting chairman of the subcommittee, and the work of the subcommittee will be forwarded during the coming year.

Subcommittee IV on Methods of Analysis (H. C. Miller, chairman).—This subcommittee is recommending that the Tentative Methods for Determination of the Calcium Ion and Magnesium Ion in Industrial Waters (D 511 – 40 T), Determination of the Chloride Ion in Industrial Waters (D 512 – 40 T), Determination of the Total Orthophosphate and Calculation of the Respective Orthophosphate Ions in Industrial Waters (D 515 – 40 T), and Determination of the Sulfate Ion in Industrial Waters (D 516 – 40 T) be continued as tentative without further revision. The subcommittee is also recommending that the Tentative Method for Determination of the Hydroxide Ion in Industrial Waters (D 514 – 38 T) be adopted as standard without revision.

Methods for the determination of silica, iron, aluminum, manganese, soap hardness, and dissolved oxygen are currently being studied by the subcommittee. It is not anticipated that these methods will be available for consideration by the Society as proposed methods for another year.

There has been a generous response to the requests for rapid routine methods, many of these are colorimetric methods and require specially calibrated color comparison standards of liquid or glass. Recent improvements in design and availability of photoelectric color comparators may make possible the elimination of such comparison standards and considerably broaden the field of usefulness of color methods in general. The subcommittee believes that it will be amply repaid if time is now taken for revision of some of these rapid routine methods to permit of their use with the commercial equipment available rather than to issue them immediately since they may then need to be extensively revised at a later date.

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part III, pp. 523, 541.

[4] 1939 Book of A.S.T.M. Standards, Part III, p. 1111.

Consideration of some of the methods mentioned in the 1940 annual report, such as procedures for determining suspended matter, organic matter, sodium, and ammonia, has been held in abeyance as the subcommittee has an exceptionally heavy schedule at present.

Subcommittee V on Classification (R. C. Adams, chairman).—Subcommittee V approved by a letter ballot, issued during the current year, the adoption as standard of the Tentative Method of Reporting Results of Analysis of Industrial Waters (D 596 – 40 T) without further revision. No other work was undertaken by this subcommittee during the year.

This report has been submitted to letter ballot of the committee which consists of 62 members; 45 members returned their ballots, of whom 39 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Secretary.

REPORT OF COMMITTEE D-20
ON
PLASTICS

Since the June, 1940, meeting in Atlantic City, N. J., Committee D-20 on Plastics and its subcommittees have held two meetings: one in New York, N. Y., in November, 1940, and one in Washington, D. C., in March, 1941. The committee expects to hold a further meeting in Chicago, Ill., during the annual meeting.

Mr. J. C. Pitzer has resigned as chairman of Subcommittee II on Hardness Properties, and W. H. Gardner has been appointed in his place.

Committee D-20 now has a membership of 72 voting members, representing an increase during the year of 7 voting members.

Subsequent to the 1940 annual meeting, Committee D-20 presented to the Society through Committee E-10 on Standards a revision of the Tentative Methods of Test for Index of Refraction of Transparent Organic Plastics. This revision was accepted[1] by Committee E-10 on August 28, 1940, and the revised methods now appear in the 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 289, under the designation D 542 - 40 T.

I. New Tentative Standards

The committee recommends that the following new proposed methods of test be accepted for publication as tentative:

Methods of:

Test for Deformation of Plastics Under Load at Elevated Temperatures.[2]

Preconditioning Plastics and Electrical Insulating Materials for Testing,[3] prepared in cooperation with Committee D-9 on Electrical Insulating Materials, and

Test for Color Fastness of Plastics to Light.[4]

II. Revisions of Tentative Standards

The committee recommends that the following tentative standards be revised as indicated below and continued as tentative:

Tentative Method of Test for Flammability of Plastics (D 568 - 40 T):[5]

Section 1.—Add the following at the end of this section:

> Two alternate methods of ignition are covered as follows:
> *Method A.*—Fusee Method, and
> *Method B.*—Benzol Drop Method.

Section 2.—Add a new Paragraph *(d)* to read as follows:

> *(d) Benzol.*—Ordinary laboratory grade benzene (C_6H_6).

Section 4.—Add a new italic heading at the beginning of Paragraph *(a)* to read "Fusee Method" and add a new sentence at the end of this paragraph to read as follows:

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee D-20 reported results of the letter ballot vote as follows: Of a total membership of 64, 47 members returned their ballots, of whom 34 voted affirmatively, 1 negatively, and 12 marked their ballots "not voting."

[2] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 440.

[3] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 320.

[4] This method was accepted as tentative by the Society and appears in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 430.

[5] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 284.

> The square of fusee material shall be ignited by means of a safety match.

Substitute for the first sentence of Paragraph (*b*) a new paragraph to read as follows:

> (*b*) *Benzol Drop Method.*—A drop of benzol shall be placed on the test specimen approximately ½ in. above the lower edge of the specimen, and allowed to run down to form a large drop on the blunt end without dripping off. This drop shall be ignited within 7 sec. after application to the test specimen with either of the following means: (*1*) A high potential, low energy spark such as delivered by an automobile ignition coil, or (*2*) A safety match. After ignition, the door of the shield shall be closed immediately (Note).

Section 5.—Add a new item 4 to be reported to read as follows: "(*4*) Method of ignition."

Tentative Method of Measuring Flow Temperatures of Thermoplastic Molding Materials (D 569 – 40 T):[6]

Section 5 (e).—Change the second sentence to read as follows by the addition of the italicized figures and the omission of those in brackets:

> Temperature shall be controlled within plus or minus [0.10] *0.50* C.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Item	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE STANDARDS			
Test for Deformation of Plastics Under Load at Elevated Temperatures	37	3	9
Preconditioning Plastics and Electrical Insulating Materials for Testing	37	3	9
Test for Color Fastness of Plastics to Light	40	1	7
II. REVISIONS OF TENTATIVE STANDARDS			
Test for Flammability of Plastics (D 568 – 40 T)	41	1	7
Measuring Flow Temperatures of Thermoplastic Molding Materials (D 569 – 40 T)	41	0	8

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 67 voting members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (M. H. Bigelow, chairman).—This subcommittee has completed its study of the tensile strength test method and the impact strength methods which have been submitted to letter ballot of the subcommittee. These methods will be referred to Committee D-9 on Electrical Insulating Materials for coordination with the Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 39) and the Standard Methods of Test for Impact Resistance of Electrical Insulating Materials (D 256 – 38).[7]

Subcommittee II on Hardness Properties (W. H. Gardner, chairman).—In addition to a continuation of the study of mar, scratch, and abrasion resistance of plastics, the subcommittee is undertaking the study of plastic bearing materials and their frictional properties, with a view to developing test methods.

The subcommittee has also prepared a new proposed Tentative Method of Test for Deformation of Plastics Under Load at Elevated Temperatures which is being submitted to the society for publication as tentative, as appended hereto.[2]

Subcommittee III on Thermal Properties (W. A. Zinzow, chairman).—This subcommittee has continued its activities in the study of the Tentative Method of Test for Measuring Flow Temperatures of Thermoplastic Molding Materials (D 569 – 40 T), also procedures for the rate of burning, and softening point of plastics. A revision has been prepared for Tentative Method D 569 in order to broaden the temperature control from plus or minus 0.10 C. to plus or minus

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[6] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 286.

[7] See Editorial Notes, pp. 397 and 474.

0.50 C. In addition, a revision adding a benzol drop method of ignition to the Tentative Method of Test for Flammability of Plastics (D 568 – 40 T) has also been completed. Those recommendations are referred to earlier in this report.

Subcommittee IV on Optical Properties (W. F. Bartoe, chairman).—A glossary of optical terms relating to plastics is in preparation. Round-robin tests are being conducted on specimens in accordance with proposed methods of test for clarity, test for surface brightness, and test for surface irregularities.

Subcommittee V on Permanence Properties (G. M. Kline, chairman).—A new proposed Tentative Method of Test for Color Fastness of Plastics to Light is being submitted to the Society for publication as tentative, as appended hereto.[3] Studies on evaluation of the weathering resistance of plastics, resistance of plastics to heat, permeability of water vapor by plastics, and simulation of service conditions are being conducted which, it is expected, will lead to the development of methods of test.

Committee D-20 has approved the definition of "dry room conditions" as 15 per cent relative humidity at 85 F. and "moist room conditions" as 75 per cent relative humidity at 77 F. The committee has also approved the establishment of a standard laboration atmosphere for testing plastics of 50 ± 2 per cent relative humidity at 77 ± 2 F.

This subcommittee has approved the Proposed Method of Preconditioning Plastics and Electrical Insulating Matetials for Testing being submitted as tentative, as appended hereto.[4] This method was prepared in cooperation with Committee D-9 on Electrical Insulating Materials and has been submitted to letter ballot of both committees.

This report has been submitted to letter ballot of the committee which consists of 67 voting members; 48 members returned their ballots, of whom 45 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. E. EMLEY,
Chairman.

W. A. EVANS,
Secretary

EDITORIAL NOTE

Subsequent to the annual meeting, Committee D-20 presented to the Society through Committee E-10 on Standards the following recommendations:

New Tentative Methods of:

Test for Flammability of Plastics, Over 0.050 in. in Thickness (D 635 – 41 T),

Test for Diffusion of Light by Plastics (D 636 – 41 T),

Test for Surface Irregularities of Flat Transparent Plastic Sheet (D 637 – 41 T),

Tension Testing of Plastics (D 638 – 41 T),

Revision of Tentative Method of:

Test for Resistance of Plastics to Chemical Reagents (D 543 – 39 T), and

Tentative Revision of Standard Methods of:

Test for Impact Resistance of Electrical Insulating Materials (D 256 – 38).

These recommendations were accepted by Committee E-10 on August 25, 1941, and the new tentative methods were assigned the A.S.T.M. serial designations indicated above. The new and revised tentative standards as well as the tentative revision appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part III, pp. 447, 433, 455, 457, 452, and 339.

REPORT OF COMMITTEE E-1

ON

METHODS OF TESTING

While Committee E-1 on Methods of Testing has not met during the year, a number of the technical committees and their sections have been active and held meetings in Washington, D. C., in March, 1941, at the time of the spring group committee meetings. Meetings of Committee E-1 and a number of its sections will be held in Chicago, Ill., during this annual meeting at which time action will be taken on various recommendations to be presented to the Society through Committee E-10 on Standards. These recommendations are referred to later in this report.

The Executive Committee of the Society appointed three representatives-at-large to serve on the Advisory Committee, namely, E. P. Partridge, Hall Laboratories, Inc., A. J. Phillips, American Smelting and Refining Co., and L. J. Trostel, General Refractories Co., each for a three-year term, expiring in 1943.

Hydrogen Ion Determinations.—Arrangements have been made for the organization of a new Technical Committee XIII on Hydrogen Ion Determinations authorized last year by the Advisory Committee on the recommendation of the Executive Committee of the Society. This meeting will be held in Chicago, Ill., during the 1941 annual meeting under the chairmanship of E. B. Ashcraft, In Charge, Micro-chemical and Spectrographic Laboratory, Westinghouse Research Laboratories. At this meeting opportunity will be provided for a general discussion of the problems involved, looking toward the formulation of a program of work. The measurement of pH value is widely used in the control of chemical and industrial processes, and it is considered one of the significant items in the study of corrosion of metals exposed to either atmospheric or sub-aqueous environments. In view of the increasing interest in the proper significance and interpretation of the pH value of a solution, the initiation of work on this subject would appear to be particularly timely.

The personnel of the committee at present includes the following representatives designated by A.S.T.M. standing committees:

W. D. Appel, representing Committee D-13 on Textile Materials,
P. H. Dike, representing Committee D-9 on Electrical Insulating Materials,
W. E. Emley, representing Committees D-6 on Paper and Paper Products and D-20 on Plastics,
M. S. Kantrowitz, representing Committee D-6,
H. C. Miller, representing Committee D-19 on Water for Industrial Uses,
Paul Rapp, representing Committee D-18 on Soils for Engineering Purposes,
H. E. Smith, representing Committee D-1 on Paint, Varnish, Lacquer, and Related Products,
W. P. Tyler, representing Committee D-11 on Rubber Products, and
B. S. Van Zile, representing Committee D-12 on Soaps and Other Detergents.

In addition to the above, special advisory members and others especially qualified by experience in pH measurements will be included in the personnel of the committee.

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing (H. F. Moore, chairman):

Herewith is presented a brief summary of the work of the various sections of this technical committee during the past year.

Section on Tension Testing (R. L. Templin, Chairman).—This section held a meeting on March 5, 1941, at which consideration was given to the criticisms which have been received concerning the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T). The section has under consideration methods for testing material less than 0.010 in. thick and material less than $\frac{3}{4}$ in. wide, as well as round specimens larger in diameter than the $\frac{1}{2}$-in. diameter size. The effects of the relation of the gage length to the cross-sectional area of the specimen upon the elongation value obtained has been studied by some of the members, and a report on "Effect of Specimen Shape and Size on Measured Values of Tensile Properties" by G. F. Jenks appears in Appendix I to this report.

The section will welcome any constructive criticism of the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T), as it desires to advance the tentative methods to standard as soon as such action is justified.

Section on Compression Testing (M. F. Sayre, chairman).—This section is considering the revision of the Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T). Further consideration will be given to this at a meeting of the section to be held during the annual meeting, following which it may be possible to present a recommendation to the main committee for submission to the Society.

Section on Indentation Hardness (J. R. Townsend, chairman.—This section held a meeting during the group meetings in Washington, D. C., this year. At that time, the holding of a Symposium on Hardness Testing at the 1941 annual meeting of the Society was considered. It was found that owing to the great pressure of work due to the Defence Program it was not feasible to secure persons to present the desired papers at that meeting.

At the meeting in Washington the matter of correlation of several types of hardness tests was discussed and data on this subject are being collected by the section. It is felt that some further revision may be necessary in the Tentative Method of Test for Rockwell Superficial Hardness of Metallic Materials (E 18 – 39 T), and it is accordingly recommended that it be continued in the tentative status.

Section on Flexure Testing (H. F. Clemmer, chairman).—This section has been considering an extensive tabulation of all flexure tests now in the A.S.T.M. standards and plans to hold a meeting during the annual meeting in Chicago to formulate a program of work based on this tabulation.

This section has been very much interested in the studies being made at the National Bureau of Standards as reported in the paper by John Tucker, Jr., on "Statistical Theory of the Effect of Dimensions and of Method of Loading Upon the Modulus of Rupture of Beams,"[1] to be presented at this annual meeting.

Section on Impact Testing (W. W. Werring, chairman).—During the past year there has been formulated a proposed revision of the Tentative Methods of Impact Testing of Metallic Materials (E 23 – 34 T). This revision was approved by letter ballot of the Impact Section and by Technical Committee I on Mechanical Testing. The proposed tentative methods have been submitted to Committee E-1 and will be considered by the committee at its meeting during this annual meeting. After approval by letter ballot the revised methods will be presented to the Society through Committee E-10 on Standards for publication this year as tentative.

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[1] See p. 1072.

Section on Effect of Speed of Testing (P. G. McVetty, chairman).—This section has been considering the extensive discussion of the effect of speed of testing which was held at the 1940 meeting. It seems evident that some specification other than that of speed of head of testing machine must be used to specify speed of testing. In the paper on "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals,"[2] by Paul G. Jones and H. F. Moore presented at the 1940 annual meeting, a method for estimating rate of strain was discussed. Such a method is suggested by these same authors in a report on "Effect of Rate of Strain on Yield Strength, Tensile Strength, Elongation, and Reduction of Area in Tension Tests" which appears in Appendix II to this report. This method does not involve the use of special apparatus other than an extensometer and a stop watch. It is a modification of a method suggested by M. O. Withey of the University of Wisconsin in connection with the report of the Research Committee on Yield Point of Structural Steel which finished its work some years ago.

Section on Elastic Strength (F. B. Seely, chairman).—This section has been concerned with the significance of limits of deviations, either involving small permanent set or slight deviation from Hooke's law—deviations smaller than those considered significant for determining "yield strength." It has also been considering the use of the term "proof stress," which occasionally is found in specifications and which seems to be used with several different meanings. At the present time the section is trying to formulate definitions on these matters which will be definite and which will involve a minimum of conflict with definitions now in actual use.

Section on Calibration of Testing Machines and Apparatus (H. F. Moore, chairman).—This section held a meeting during the spring group meetings in Washington. At that time it considered the first draft of proposed tentative standard for the calibration of extensometers. This draft was, in general, approved by those present, but certain minor revisions seemed desirable and the proposed tentative standard is to be submitted to letter ballot of the section when the revisions are completed. In considering the calibration of extensometers, the section has an advantage of being in an unworked field so that it is hoped that it may formulate standards without conflict with standards already in use.

Technical Committee II on Consistency, Plasticity, and Related Properties (Eugene C. Bingham, chairman):

The revised Tentative Definitions of Terms Relating to Rheological Properties of Matter (E 24 - 40 T) are now before the Society in tentative form. They have been the subject of discussion for from five to ten years as they have been elsewhere, as in the Society of Rheology. This discussion is valuable, particularly in stimulating research. The Society of Rheology has adopted working definitions but there is no thought of anything approaching finality.

It is significant to report active work of members of this Society directed toward the more exact measurement of the flow of engineering materials and the correlation of the many flow tests now being used in industry. One of the first steps has been to determine whether the results of a given test are a simple or a complex function of the fluidity, possibly containing other parameters, such as the *yield value*, *elasticity*, etc.

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 610 (1940).

This committee at its meeting in June, 1940, appointed a Special Research Committee on Industrial Flow Tests under the chairmanship of Robert Burns, Bell Telephone Laboratories, to devise a plan for accelerating this fundamental work. The special committee decided to request a small grant of money from the A.S.T.M. Research Fund to facilitate the initiation of the project. This request was granted by Committee E-9 on Research and the sum of $500 has been placed at the disposal of the committee. The money is being used in a preliminary investigation, in the hope that results may be obtained which will justify a further and more extensive investigation. Because of its wide use and its favorable showing, the ring-and-ball softening point test was selected for study, comparing results obtained with it with those obtained with various types of viscosimeters, of which the capillary tube type and the rotating cylinder type are familiar.

This work is already under way in the recently established Rheological Laboratory at the Brooklyn Polytechnic Institute by H. Mark under the supervision of W. H. Gardner, in charge of the department and a member of the special committee.

The above work on a single technical test is purposely a very limited project in order that measurable results may be obtained with limited effort. The larger problem of measuring the flow in absolute units of certain types of colloids, and of metals and their alloys is far more baffling and may require much patient effort.

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

A meeting of the Section on Pigment-Type Materials (C. E. Barnett, chairman) was held in April, 1940, in Cincinnati, Ohio, at which time the experimental background of the original and the work on the revision of the Tentative Method of Test for the Particle Size Distribution of Subsieve Size Particulate Substances (E 20 – 33 T) was discussed. It was concluded that the present state of knowledge did not justify retaining the method as tentative. A cooperative program was outlined to study some of the points which have been raised in the literature against the fundamental principles involved in size measurements of particulate materials with the microscope. This program is making fair progress and while the section is not yet ready to report on the work, it feels that results so far are sufficiently encouraging to justify continuing Method E-20 in its present status.

Under the sponsorship of this section a Symposium on New Methods for Particle Size Determinations in the Subsieve Range was held in Washington, D. C., on March 4, during the 1941 spring meetings. The symposium included seven papers, as follows:

"The Shape and Surface of Fine Powders by the Permeability Method," by P. C. Carman, University of Capetown,

"Surface Area of Portland Cement," by P. S. Roller, U. S. Bureau of Mines and P. V. Roundy, Jr., American Instrument Co.,

"Methods for Determining Particle Size Distribution," by Lincoln T. Work, Metal and Thermit Corporation and Herbert E. Schweyer, Columbia University,

"RCA Electron Microscope," by James Hillier, RCA Manufacturing Company,

"The Absorption of Pigments," by W. W. Ewing, Lehigh University,

"A New Method for Measuring the Surface Areas of Finely Divided Materials and Its Use in Determining the Size of Particles," by P. H. Emmett, Johns Hopkins University, and

"The Determination of Subseive Particle Size Distributions by Sedimentation Methods," by S. W. Martin, National Lead Company.

The Section on Sieves (A. A. Klein, chairman) and the Section on Ground Products (L. T. Work, chairman) are merely watching developments and contemplate no immediate efforts in the development of new standards.

Technical Committee IX on Interpretation and Presentation of Data (H. F. Dodge, chairman):

During the past year this technical committee has continued to serve in an advisory capacity to several standing committees of the Society on problems relating to the use of statistical methods in the analysis and presentation of data. The Manual Committee has made some progress in a revision of the A.S.T.M. Manual on Presentation of Data. This revision will be in the nature of an additional supplement dealing with the question of sampling errors of averages. As this work had not been completed and since the available stock of the August, 1940, printing of the Data Manual was exhausted early in 1941, the manual was again reprinted in March, 1941. In this reprinting the opportunity was taken to correct two values in Table I of Supplement B which were in error in the August, 1940, printing. Permission was granted to Emergency Technical Committee Z-1 on Quality Control to reproduce Supplement B of the A.S.T.M. Data Manual as an appendix to the Proposed American Defense Emergency Standard Control Chart Method of Analyzing Data, being prepared under the auspices of the American Standards Association.

Technical Committee X on Conditioning and Weathering (Robert Burns, chairman):

In May, 1940, this technical committee was expanded in scope and personnel to cover activities in weathering as well as conditioning fields under the new title given above. Several standing committees not previously represented have appointed members to serve on the expanded committee.

This committee has maintained close contact with all standing committees concerned with conditioning and weathering and has rendered advice and assistance. In several instances duplication has been avoided and in others valuable information has been interchanged between committees as well as with other engineering societies.

In accordance with the committee's policy of standardizing important items in the field of atmospheric influences, the members are now reviewing proposed Tentative Definitions, Tables, and Related Items in Conditioning and Weathering. The purpose of this method is to obtain Society-wide recognition and use of any items originated by standing committees which are broadly applicable to industry as a whole. Upon such a method and future revisions thereof the materials testing technician can rely for a comprehensive picture of the Society's accomplishment in this field. The proposed definitions contain the following:

1. Definition of "Conditioning," prepared by Technical Committee X,
2. Definition of "Average Room Conditions," prepared by Committee D-9 on Electrical Insulating Materials,
3. Definition of "Dry Room Conditions," prepared by Committee D-9 and Committee D-20 on Plastics,
4. Definition of "Moist Room Conditions," prepared by Committees D-9 and D-20,
5. Psychrometric methods, definitions, and tables, prepared by Committee D-13 on Textile Materials,
6. Determination of room temperature, prepared by Committee D-9,
7. Establishment of standard laboratory atmosphere, prepared by Committees D-9 and D-20, and
8. Appendix of useful information.

Technical Committee XI on Designation and Interpretation of Numerical Requirements (J. A. Gann, chairman):

This technical committee has held no meetings since March 6, 1940, at which time it completed the preparation of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (E 29 - 40 T) which were accepted by the Society at the 1940 annual meeting for publication as tentative.

The committee trusts that these tentative recommended practices will be carefully studied by the standing committees and will be used by them in the preparation of A.S.T.M. specifications. Such use will demonstrate the practicability of these recommended practices, as well as disclose any shortcomings in them. The committee has no immediate program on its docket but is awaiting an evaluation of the recommended practices by the various committees of the Society.

Technical Committee XII on Laboratory Apparatus (W. H. Fulweiler, chairman):

A meeting of this technical committee and several of its sections was held in Washington, D. C., on March 5, 1941.

In view of the variety of apparatus now specified in A.S.T.M. standards, and the increasing additions resulting from the development of new apparatus in the preparation of new methods of test, it has been considered desirable to have available detailed information as to the sources of special apparatus prescribed in Society standards. In this endeavor the Scientific Apparatus Makers of America is cooperating.

The technical committee, after reviewing the form in which dimensional requirements for laboratory apparatus appear in A.S.T.M. standards, has indicated that, in general, use should be made of dimensional drawings showing permissible variations and significant details of laboratory apparatus in preference to including such information in the text.

At the meeting of the technical committee held during this annual meeting action was taken to present to Committee E-1, for submission to the Society, a resolution[3] concerning the matter of priorities during the National Emergency for the securing of critical materials required for use in manufacturing scientific laboratory apparatus.

Section A on Thermometers (R. M. Wilhelm, chairman).—This section has been very active during the year. At the March meeting in Washington agreement was reached on detailed specification requirements for the titer test thermometer. These specifications were prepared in cooperation with the American Oil Chemists Society, and A.S.T.M. Committee D-12 on Soaps and Other Detergents. These specifications, as appended hereto[4] as information, have been submitted to Committee E-1 for approval at the June meeting following which they will be presented to the Society for publication as tentative through Committee E-10 on Standards.[5]

The section has also recommended that the specifications for the three aniline point thermometers, appended hereto as information,[3] be published as tentative. The requirements for these thermometers have been prepared in cooperation with Committee D-2 on Petroleum Products and Lubricants and are required for use in connection with the proposed Tentative Method of Test for Aniline Point of Petroleum Products.[6] Studies now under way at the National Bureau of Standards on the average equivalent stem temperatures of this thermometer are expected to result in a further revision of these specifications. It is expected that this information will be available at the June meeting for inclusion in the specifications which will be acted on at that time for submission to the Society as tentative through Committee E-10.[5]

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[3] This resolution as adopted by the Society appears in the Summary of Proceedings, see p. 30.

[4] These specifications were accepted as tentative by the Society and appear in the 1941 Supplement to Book of A.S.T.M. Standards, Parts II and III.

[5] See Editorial Note, p. 482.

[6] 1941 Supplement to Book of A.S.T.M. Standards, Part III, p. 257.

The section has recommended the following note for inclusion in connection with the tung oil and turpentine distillation thermometers, and the thermometer for stability test of soluble nitrocellulose appearing in Specifications E 1 - 40 T and E 1 - 39, respectively:

> NOTE.—Due to the application requirements for range and construction of this thermometer, it is not practicable to include reference points. Therefore, the National Bureau of Standards will issue a report instead of a certificate for this instrument.

A revision has been prepared in the requirements for the A.S.T.M. high distillation thermometer E 1 (8C - 39) and E 1 (8F - 39) as prescribed in the Standard Specifications for A.S.T.M. Thermometers (E 1 - 39). The proposed revision will change the distance from the bottom of the bulb to the graduation line at 0 C. or 32 F. from the present specified dimension of "25 to 35 mm. (0.98 to 1.38 in.)" to a distance of "35 to 45 mm. (1.38 to 1.77 in.)." This proposed revision is being published at this time only as information. The committee plans to recommend this change for formal adoption as standard by the Society at the 1942 annual meeting to become effective, if approved, as of December 1, 1942.

A revision has also been proposed by the committee in the requirements for the A.S.T.M. open flash thermometer E 1 (11C - 39) and E 1 (11F - 39). This proposed revision will reduce the length of the bulb from the present specified requirement of "not over 13 mm. (0.51 in.)" to read "not over 10 mm. (0.39 in.)." This change is also published as information and will be presented to the Society at the 1942 annual meeting to become effective, if approved, as of December 1, 1942.

The committee has also approved for publication as information in this report the following note with reference to the standardization of the A.S.T.M. kinematic viscosity thermometer covered by the Tentative Specifications for A.S.T.M. Thermometers (E 1 - 40 T):

> Two methods have been in use for determining the ice point on thermometers graduated fractionally and with an open scale such as those of the Kinematic type. One method requires that the thermometer remain at approximately room temperature for 72 hrs. before the ice point is taken. The other stipulates that the ice point reading be taken immediately or within a definitely stated short period after heating to the test temperature. Changes in the correction of the thermometer may be observed by determining the changes in the ice point by either method.
>
> Since it may not be practical to wait until the thermometer has rested for three or more days, the method as outlined below was suggested. These instructions are given in the form of a note which should appear in the table of corrections for the thermometer.
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> NOTE.—The tabulated corrections apply for the condition of immersion indicated provided the ice point reading taken after heating to . . . . . . for not less than three minutes is . . . .
>
> If the ice point reading (taken in not less than 2 min. and not more than 5 min. after removal of the thermometer from the heated bath) is found to be higher (or lower) than stated, all other readings will be higher (or lower) to the same extent.

Consideration is being given, in cooperation with the National Bureau of Standards, to ice point determinations on A.S.T.M. kinematic viscosity thermometers. A study is being made of the desirability of limiting the maximum bulb length for the A.S.T.M. open flash thermometer. The section has received a request from Committee D-3 on Gaseous Fuels in connection with specifications for calorimetric thermometers for flow calorimeters.

Section B on Hydrometers (J. P. Bader, chairman).—The chairman of this section in cooperation with a representative from the National Bureau of Standards has in preparation a paper on "Testing of Hydrometers" for publication as an appendix to the 1942 report. In addition to discussing in detail methods of testing hydrometers, the paper will discuss the origin and history of hydrometer scales, particularly the A.P.I. scale; also various arbitrary scales and formulas for calculating specific gravity equivalents and *vice-versa.*

Section C on Volumetric Glassware (J. J. Moran, chairman).—A paper on "Methods of Testing Volumetric Glassware" prepared by the chairman of this section appears as Appendix III to this report.

This section has during the year continued to cooperate with several of the standing committees in reviewing copies of new and revised methods containing requirements for calibrated glass apparatus.

Section D on Laboratory Glassware (F. F. Shetterly, chairman).—This section has reviewed and proposed improvements in requirements for certain pieces of laboratory glassware appearing in several existing A.S.T.M. methods. These recommendations have been submitted to the standing committees responsible for the methods in question.

Section E on Ceramic Apparatus (H. W. Ryland, chairman) *and Section F on Metalware Apparatus* (J. E. Patterson, chairman).—These sections which were organized last year are prepared to serve in an advisory capacity to the standing committees on any matters dealing with questions covering ceramic and metalware apparatus that may be developed in connection with A.S.T.M. test methods.

This report has been submitted to letter ballot of the committee which consists of 52 members; 29 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

P. J. SMITH,
Ex-officio Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting, Committee E-1 presented to the Society through Committee E-10 on Standards the following recommendations:

Revision of Tentative:

Methods of Impact Testing of Metallic Materials (E 23 – 40 T),
Specifications for A.S.T.M. Thermometers (E 1 – 40 T), and

Tentative Revision of Standard:

Specifications for A.S.T.M. Thermometers (E 1 – 39).

These recommendations were accepted by Committee E-10 on August 25, 1941, and the revised tentative standards and tentative revision appear in the 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 572, Part II, pp. 409 and 426, and Part III, pp. 619 and 639.

APPENDIX I

EFFECT OF SPECIMEN SHAPE AND SIZE ON MEASURED VALUES OF TENSILE PROPERTIES[1]

By G. F. Jenks[2]

Three brands of low-alloy steel plates of five thicknesses were tested using round and flat specimens. Both longitudinal and transverse properties were measured for three thicknesses and longitudinal properties only for two thicknesses.

The round specimens used were 0.357 in. in diameter for plates $\frac{3}{8}$ in. and greater in thickness, 0.252 in. in diameter for plates 0.345 in. thick and 0.178 in. in diameter for plates 0.210-in. thick. In all cases the gage length was equal to $4.5\sqrt{A}$, where A is the area of the test specimen.

Two series of flat specimens were used, one 0.75 in. and the other 1.5 in. in width. The gage section was tapered toward the center by approximately 0.030 in. in order that the break would occur at a point midway between the gage marks. The gage lengths used were 1.0 in., 2.0 in., 4.0 in., and the length calculated from the ratio $L = 4.5\sqrt{A}$.

The data obtained in the tests are shown in Tables I, II, and III. Each value given is the average of three values, three specimens being tested for each condition. The variations between specimens were normal for uniform material. The tables indicate the brands and thicknesses used.

Discussion:

The yield strength (0.1 per cent offset) in comparison with that from round specimens was found to average 186 psi. less on the 0.75-in. flat specimen and 1882 psi. less on the 1.5-in. flat specimen.

There was no appreciable difference in yield strength for specimens taken longitudinally and transversely.

The variations in measured tensile strength were much greater. There was no appreciable difference in values for the two widths of flat specimens. The results obtained with flat specimens were about 5 per cent higher in the case of the $\frac{5}{8}$-in. plates but were lower for the other thicknesses. The average variation for all thickness is small for both widths of specimens.

For different shapes and sizes of specimens in which $L = 4.5\sqrt{A}$, the average variations in elongation are not appreciable. The 0.75-in. specimen averaged 0.05 per cent greater and the 1.5-in. specimens averaged 0.02 per cent less elongation than the round specimens. The extreme variation in elongation was 0.4 per cent or 1.6 per cent of the measured elongation. This percentage variation is about one-third of the extreme percentage variation in tensile strength and occurred in the same material.

Of greater interest is the wide spread of elongation values for various gage

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[1] Publication authorized by the War Department.

[2] Colonel, Ordnance Dept., U. S. Army.

TABLE I.—COMPARATIVE TENSILE PROPERTIES FOR ROUND AND FLAT TEST SPECIMENS, ROLLED PLATE.

Grade	Thickness, in.	Direction of Fibers	Yield Strength (0.1 per cent Offset), psi.			Tensile Strength, psi.		
			Round Specimens	Flat Specimens		Round Specimens	Flat Specimens	
				0.75-in. Wide	1.5-in. Wide		0.75-in. Wide	1.5-in. Wide
Navy HT	⅝	Longitudinal	66 580	65 830	63 500	91 860	96 300	96 000
		Transverse	65 580	66 280	63 700	91 460	96 630	96 300
Yoloy	½	Longutidinal	64 500	64 000	62 000	93 900	92 700	91 760
		Transverse	65 500	65 000	61 000	93 700	92 800	91 430
Navy HT	⅜	Longitudinal	54 080	54 160	54 750	80 260	77 630	77 430
		Transverse	53 920	53 000	53 920	80 060	76 600	76 930
Nat. Tube	0.345	Longitudinal	80 200	81 400	....	116 400	114 900	....
Nat. Tube	0.210	Longitudinal	80 800	80 000	....	114 400	111 500	....

TABLE II.—ELONGATION AS A FUNCTION OF $\frac{L}{\sqrt{A}}$ FOR VARIOUS COMPOSITIONS, PLATE THICKNESSES, SPECIMEN SIZES, AND DIRECTION OF FIBER.

Grade	Thickness, in.	Flat Specimen 0.75-in. Wide		Flat Specimen 1.5-in. Wide		Round Specimens		
						0.357-in. Diam.	0.252-in. Diam.	0.178-in. Diam.
		Gage Length, in. $\frac{L}{\sqrt{A}}$	Elongation, per cent	Gage Length, in. $\frac{L}{\sqrt{A}}$	Elongation, per cent	Elongation, per cent	Elongation, per cent	Elongation, per cent
Navy HT Longitudinal	⅝	1.5	55.3	1.03	62.3	....	....	....
		2.9	34.3	2.06	41.8	....	....	....
		....	....	4.12	25.5	....	....	....
		4.5	25.7	4.5	24.9	25.3	....	....
		5.8	21.3	....	....	....	....	....
Navy HT Transverse	⅝	1.5	47.7	1.03	57.3	....	....	....
		2.9	30.7	2.06	38.2	....	....	....
		....	....	4.12	24.0	....	....	....
		4.5	23.1	4.5	23.3	23.0	....	....
		5.8	19.6	....	....	....	....	....
Yoloy Longitudinal	½	1.6	45.0	1.15	48.7	....	....	....
		3.2	30.3	2.3	36.3	....	....	....
		4.5	24.3	4.5	24.4	24.1	....	....
		6.4	20.3	....	....	....	....	....
Yoloy Transverse	½	1.6	42.0	1.15	43.0	....	....	....
		3.2	27.4	2.3	32.2	....	....	....
		4.5	22.2	4.5	22.1	22.1	....	....
		6.4	19.6	....	....	....	....	....
Navy HT Longitudinal	⅜	1.9	49.3	1.33	60.0	....	....	....
		3.8	32.7	2.67	41.8	....	....	....
		4.5	29.4	4.5	29.3	29.6	....	....
		7.6	22.5	5.33	28.3	....	....	....
Navy HT Transverse	⅜	1.9	46.2	1.33	51.7	....	....	....
		3.8	30.8	2.67	37.7	....	....	....
		4.5	27.5	4.5	27.8	27.7	....	....
		7.6	21.0	5.33	26.2	....	....	....
Nat. Tube Longitudinal	0.345	2.0	38.0	....	....	....	....	....
		3.9	23.5	....	....	....	....	....
		4.5	22.3	....	....	....	22.3	....
		7.9	19.0	....	....	....	....	....
Nat. Tube Longitudinal	0.210	2.5	32.0	....	....	....	....	....
		4.5	20.2	....	....	....	....	20.2
		5.0	19.3	....	....	....	....	....
		10.0	15.0	....	....	....	....	....

lengths and for various ratios of $\frac{L}{\sqrt{A}}$.

In Fig. 1 is plotted the elongation as a function of gage length. In the graph the solid dots represent elongation value for round specimens in which $L = 4.5\sqrt{A}$. The results obtained for 0.75-in. specimens are plotted by a single circle and those from 1.5-in. specimens by a double circle. The plotted curves indicate differences between steels.

TABLE III.—ELONGATION AS A FUNCTION OF GAGE LENGTH, AND SPECIMEN SIZE FOR VARIOUS COMPOSITIONS.

Grade	Thickness, in.	Flat Specimen 0.75-in. Wide		Flat Specimen 1.5-in. Wide		Round[b] Specimens
		Gage Length, in.	Elongation, per cent	Gage Length, in.	Elongation, per cent	Elongation, per cent
Navy HT Longitudinal	⅝	1.0	55.3	1.0	62.3	....
		2.0	34.3	2.0	41.8	....
		3.1[a]	25.7	....	....	....
		4.0	21.3	4.0	25.5	....
				4.36[a]	24.9	25.3
Navy HT Transverse	⅝	1.0	47.7	1.0	57.3	....
		2.0	30.7	2.0	38.2	....
		3.1[a]	23.1	....	....	....
		4.0	19.6	4.0	24.0	....
				4.36[a]	23.3	23.0
Yoloy Longitudinal	½	1.0	45.0	1.0	48.7	....
		2.0	30.3	2.0	36.3	....
		2.75[a]	24.3	3.92[a]	24.4	24.1
		4.0	20.3	....	....	....
Yoloy Transverse	½	1.0	42.0	1.0	43.0	....
		2.0	27.4	2.0	32.2	....
		2.75[a]	22.2	3.92[a]	22.1	22.1
		4.0	19.6	....	....	....
Navy HT Longitudinal	⅜	1.0	49.3	1.0	60.0	....
		2.0	32.7	2.0	41.8	....
		2.4[a]	29.4	3.375[a]	29.3	29.6
		4.0	22.5	4.0	28.3	....
Navy HT Transverse	⅜	1.0	46.2	1.0	51.7	....
		2.0	30.8	2.0	37.7	....
		2.4[a]	27.5	3.375[a]	27.8	27.7
		4.0	21.0	4.0	26.2	....
Nat. Tube Longitudinal	0.345	1.0	38.0	....	....	....
		2.0	23.5	....	....	....
		2.28[a]	22.3	....	....	22.3
		4.0	19.0	....	....	....
Nat. Tube Longitudinal	0.210	1.0	32.0	....	....	....
		1.8[a]	20.2	....	....	20.2
		2.0	19.3	....	....	....
		4.0	15.0	....	....	....

[a] Gage length for $\frac{L}{\sqrt{A}} = 4.5$.

[b] In the case of the round specimens, the elongation values reported were determined on specimens having gage lengths of $\frac{L}{\sqrt{A}} = 4.5$.

In Fig. 2 the same elongation values are shown as a function of the ratio $\frac{L}{\sqrt{A}}$. It will be noted that for any material the influence of specimen shape is extremely small for $\frac{L}{\sqrt{A}} = 4.5$. Unfortunately the tests with 1.5-in. specimens were not carried out for gage lengths of 8 in., the standard gage length of A.S.T.M. specimens.

For round specimens, A.S.T.M. practice specifies a fixed ratio for $\frac{L}{\sqrt{A}}$ as indicated in Section 7 of the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T).[2] For flat specimens there is no common practice in this respect. Section 4 of Methods E 8 – 40 T specifies a gage length of 8 in. irrespective of thickness for thicknesses of $\frac{3}{16}$ in. or over. Specifications, such as the A.S.T.M. Standard Specifications for Steel for Bridges and Buildings (A 7 – 39),[3] for Structural Nickel Steel (A 8 – 39)[3] for Mild Steel Plates (A 10 – 39),[3] and for Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 39),[3] authorize allowances in specified elongation values for variation in thickness. For thickness of $\frac{3}{4}$ in., in which $\frac{L}{\sqrt{A}}$ is 7.5, and for thickness of $\frac{5}{16}$ in which $\frac{L}{\sqrt{A}}$ is 11.7, the specified value of the elongation holds. When A.S.T.M. specifications provide for round tension specimens, the elongation is specified separately for that specimen.

The ratio $\frac{L}{\sqrt{A}} = 4.5$ holds for a flat specimen slightly over 2-in. thick with the 8-in gage length. For such thicknesses the round specimen would probably be used as more economical to test. The elongation correction factor calculated by the rule of Section 8 (a) in A.S.T.M. Specifications A 8 – 39 would be 5 per cent (decrease in required value). Some of the steels tested were equivalent to the grade covered by A.S.T.M. Speci-

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[2] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 453.

[3] 1939 Book of A.S.T.M. Standards, Part I.

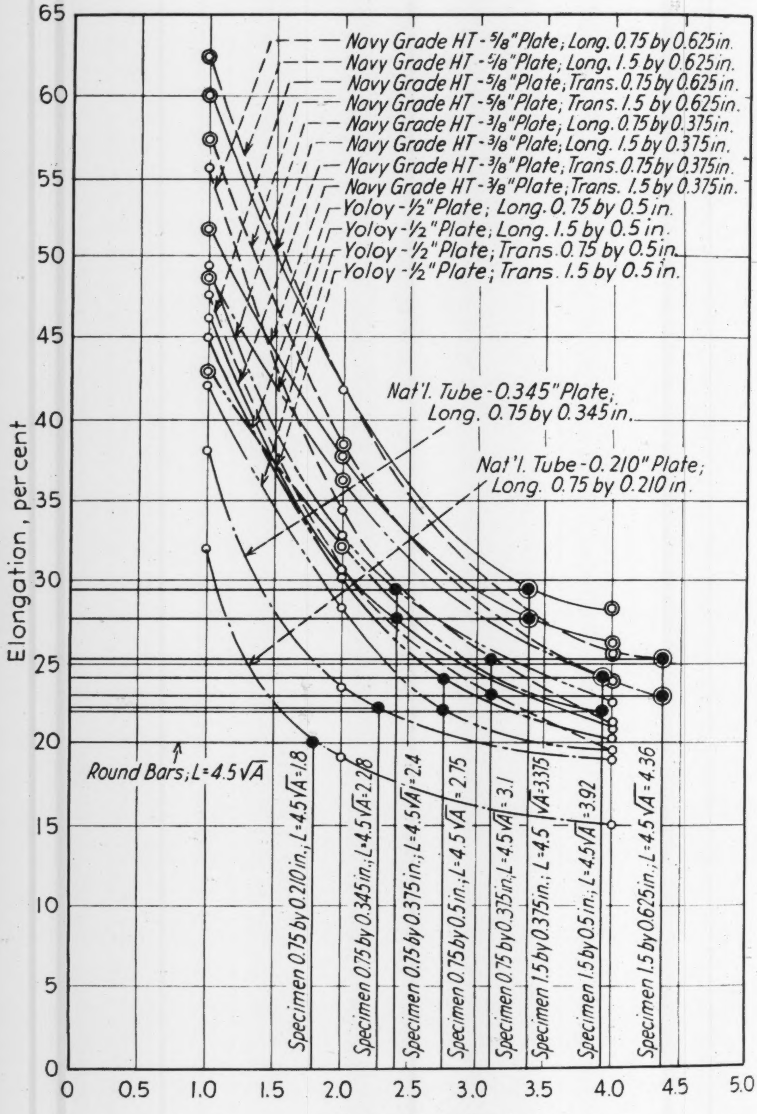

FIG. 1.—Comparison Curves Showing Elongation *versus* Gage Length. Round and Flat Specimens

fications A 203–39, where the elongation correction factor is of the same amount. The curves of Fig. 2 indicate a difference in elongation values corresponding to $\frac{L}{\sqrt{A}} = 4.5$ and those of 7.5 or greater which approximate these correction factors.

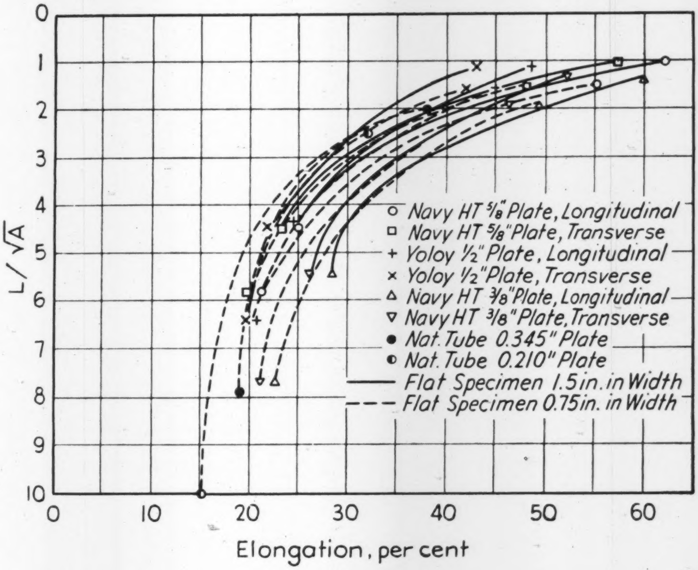

FIG. 2.—Elongation of Several Steels Measured for Various Values of $\frac{L}{\sqrt{A}}$.

It will be noted that 0.75-in. specimens are more sensitive to changes in the ratio $\frac{L}{\sqrt{A}}$ than the A.S.T.M. standard 1.5-in. specimens. It will consequently be found more difficult to judge steel quality if the narrower specimens are used.

Especially in the case of low-alloy steels it may be desired to compare quality of rolled steel with that of forged steel. In that case it is important that the ratio $\frac{L}{\sqrt{A}}$ remain constant. The A.S.T.M. flat specimens are of undesirable shape because of the large correction factor necessary to compare results with those obtained from the round test specimens used in testing forged steel.

A flat specimen with a gage length proportioned to the formula $L = 4.5\sqrt{A}$ would be satisfactory for strength and elongation measurements. If, however, reduction of area is also required the round specimen is to be preferred.

Acknowledgment:

The test data analysis was obtained by the Watertown Arsenal under the supervision of H. C. Mann, Senior Materials Engineer.

APPENDIX II

EFFECT OF RATE OF STRAIN ON YIELD STRENGTH, TENSILE STRENGTH, ELONGATION, AND REDUCTION OF AREA IN TENSION TESTS

By P. G. Jones[1] and H. F. Moore[2]

During the past year experiments have been made on five metals to see whether the effect of rate of strain can be determined without the use of the special oscillograph apparatus described in the paper by Paul G. Jones and H. F. Moore on "An Investigation of the Effect of Rate of Strain on the Results of Tension Tests of Metals."[3] The 1941 tests were made on the following five metals:

1. S. A. E. 1045 steel,
2. S. A. E. 1020 steel,
3. Hard-drawn copper,
4. Duralumin, and
5. Drill temper brass.

Specimens of all these metals were tested in the 1940 tests with the oscillograph recorder, but unfortunately it was not possible in all cases to secure specimens from the same set of bars of metal which furnished specimens for the 1940 tests. However, metal as nearly like the 1940 metal specimens as possible was obtained and results have been compared on the basis of *percentage* change in properties with change of rate of strain.

In the 1941 tests made without the use of the oscillograph, the rate of strain was determined as follows: For the slowest speed of head of testing machine the rate of strain for each lot of specimens was determined using an extensometer and a stop watch. It was then assumed that the rate of strain for other speeds of head would be proportional to the speed of head for similar specimens of the same metal. This assumption is discussed in detail in the 1940 paper.[3]

In the 1941 tests a 100,000-lb. Olsen screw-power machine was used, and tests were made at six different speeds of head. For the 1940 tests made with the oscillograph a 200,000-lb. Riehle screw-power testing machine was used with four speeds of head.

Figures 1 to 4 show the results of the 1941 tests without oscillograph and also the results of the 1940 tests with oscillograph. It will be noted that, with the exception of the yield strength of the S. A. E. 1020 steel, the percentage variation in properties for variations in rate of strain are quite closely the same when determined by the oscillograph tests and by the 1941 tests without oscillograph. Even in the case of the yield strength of the S. A. E. 1020 steel the difference in results for the two methods is not great within the usual range of speed used for determining the yield strength, say from 0.1 to 1.0 per cent strain per minute. The yield strength for this metal was taken as determined by the drop of the beam, that is, by the determination of the "upper" yield point, and the S. A. E. 1020 steel tested in the 1941 tests showed

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[1] Instructor in Theoretical and Applied Mechanics, University of Illinois.

[2] Research Professor of Engineering Materials, University of Illinois.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 610 (1940).

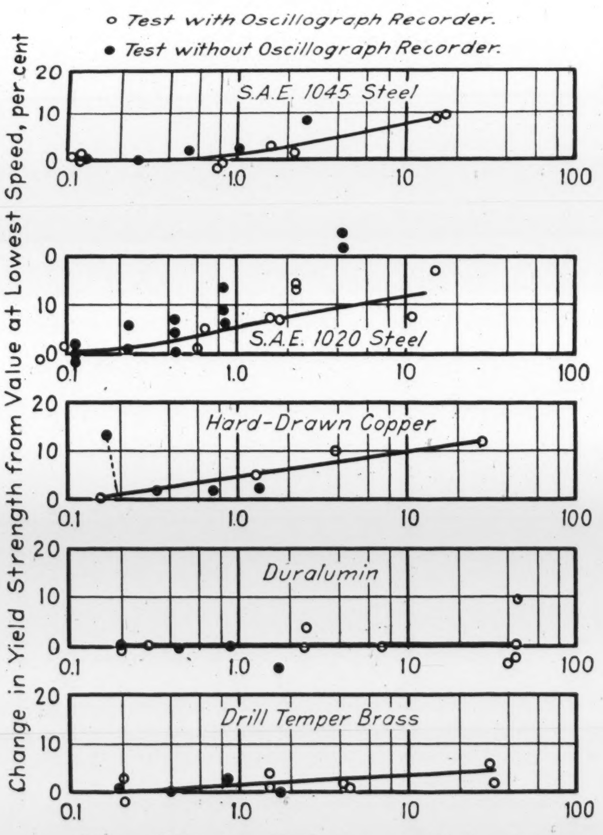

FIG. 1.—Effect of Rate of Strain on Yield Strength.

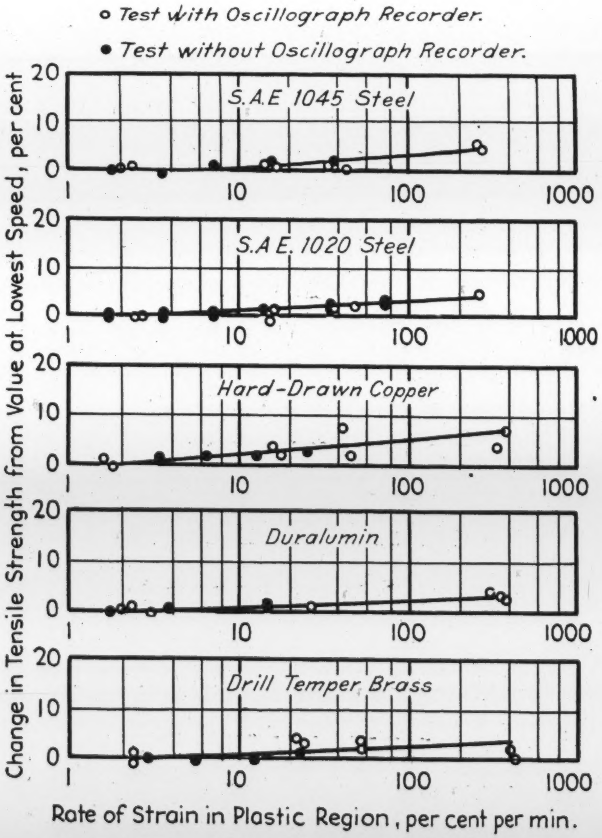

FIG. 2.—Effect of Rate of Strain on Tensile Strength.

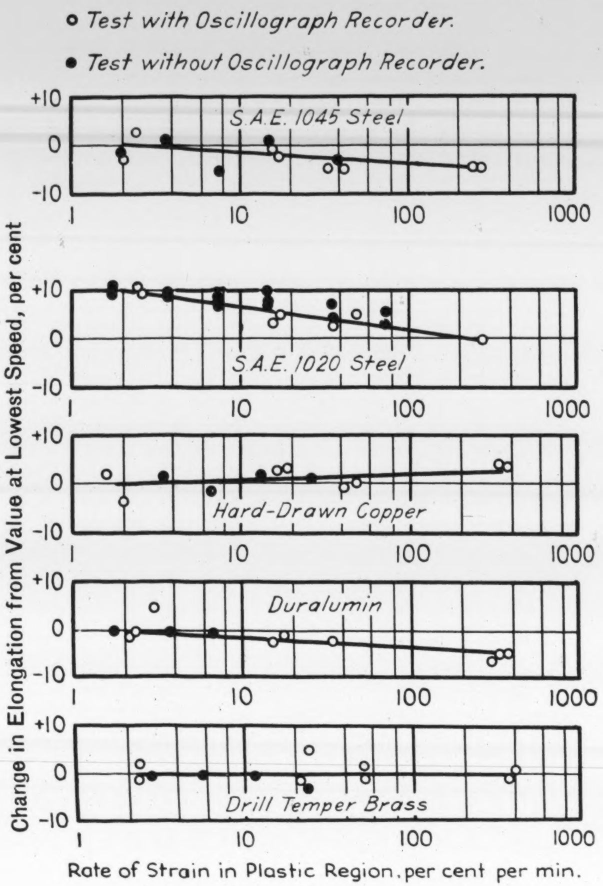

FIG. 3.—Effect of Rate of Strain on Elongation in 2 in.

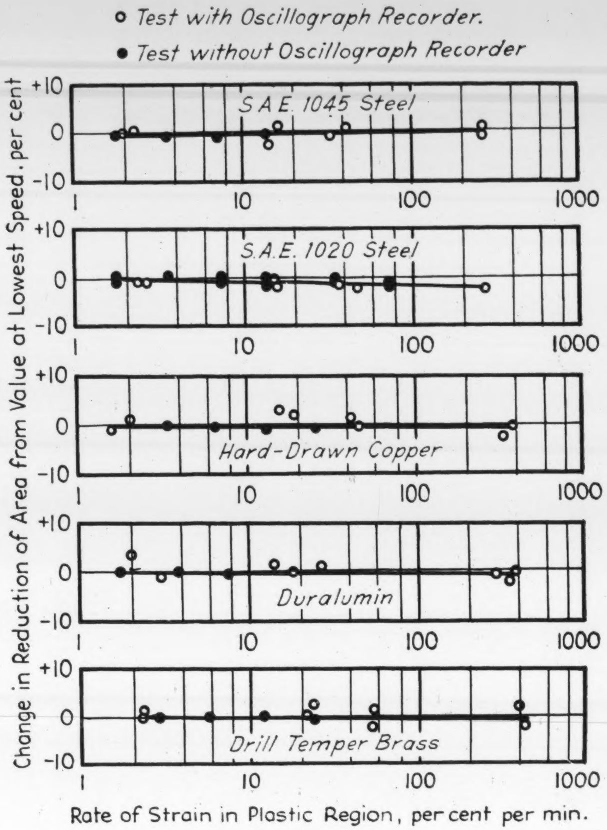

FIG. 4.—Effect of Rate of Strain on Reduction of Area.

considerable "scatter" in yield point results.

It is believed that the 1941 tests give added confidence in the reliability of determining rate of strain without the use of elaborate special apparatus. As in the 1940 paper, it may be pointed out that the method used for determining rate of strain without the oscillograph is a modification of the method proposed by M. O. Withey of the University of Wisconsin in connection with the study of the yield point of structural steel reported by the Research Committee on Yield Point of Structural Steel.[4]

Of course, the tests herein reported cover only rates of strain which can be obtained on the ordinary testing machine, and do not enter the field of study of the effect of extremely high rates of strain.

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 136 (1931), and Vol. 37, Part I, p. 87 (1937).

APPENDIX III

METHODS OF TESTING VOLUMETRIC GLASSWARE

By J. J. Moran[1]

Detailed specifications for many items of calibrated glassware designed for specific purposes appear in various A.S.T.M. Standard and Tentative Methods of Test. A wide variety of so-called "standard apparatus" is also required for various tests. Nowhere, however, are there any directions as to the methods to be used to determine the quality of any of this apparatus.

It is the purpose of this paper to give some general requirements for good apparatus and to suggest suitable methods for precise calibration. General directions only are given which can be applied to any piece the user may wish to check. No attempt has been made to give specific directions for each different item, and rapid methods suitable only for a preliminary examination of shipments will not be outlined. Much of the data presented here has been taken from publications of the National Bureau of Standards.[2]

General Requirements

Standard Temperature and Volume:

In the United States, the standard temperature for glass apparatus is 20 C. Unless some other temperature is called for in a particular specification, manufacturers will base all volumes on this temperature.

The units of volume employed in volumetric work are the liter and the one-thousandth part of a liter, called the milliliter (ml.). The liter is defined as the volume occupied by a quantity of pure water at 4 C. having a mass of 1 kg.

Annealing:

Annealing is necessary to eliminate unnecessary breakage and to minimize changes in volume which may otherwise occur with time. Different kinds of glass require different treatments to obtain satisfactory annealing. Moreover, annealing methods may change as further research is conducted. No one method of annealing, therefore, can be specified. Satisfactory apparatus should pass the following test:

"Upon examination in a polariscope there shall be no ring strain, and if there is any longitudinal strain present it should be faint and highly diffused."

The action of polarized light as a means of detecting strains in glass and the construction and use of various types of polariscopes have been discussed by Preston.[3] Since publication of his report, Polaroid instruments for this purpose also have been developed.

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[1] Technical Manager, Sales Dept., Kimble Glass Co., Vineland, N. J.

[2] N. S. Osborne and B. H. Veazey, "The Testing of Glass Volumetric Apparatus," *Bulletin*, Nat. Bureau Standards, Vol. 4, No. 4, pp. 564–595.

Anonymous, "Testing of Glass Volumetric Appartus," *Circular No. 9*, Eighth Edition, Nat. Bureau Standards (1916).

[3] F. W. Preston, "The Use of Polariscopes in the Glass Industry," *Journal*, Am. Ceramic Soc., Vol. 3, No. 9, pp. 595–623 (1930).

Design and Workmanship:

Detailed requirements for precision volumetric glassware have been published by the National Bureau of Standards.[2] These regulations should be consulted when inspecting such apparatus.

For general laboratory use, particularly in control work, some relaxation of the requirements can be permitted. The changes, however, should be limited to details such as style of lining and time of delivery of contents, and should not extend to the quality of workmanship or the general rules governing good design. The effect of such changes on the utility of the apparatus must be kept in mind, both when setting up specifications for new items and when inspecting apparatus made according to such specifications. For example, larger errors in capacity will be found when diameters at calibration points are increased or time of delivery is decreased.

SPECIAL NOTES ON MANIPULATION

Cleanliness of Apparatus:

Certain contaminations, especially grease, adhering to the walls of measuring vessels prevent them from being uniformly wetted. The tendency of water to collect into drops instead of adhering to the glass surface as a continuous film indicates imperfect wetting due to uncleanliness.

Imperfect wetting causes irregularities in capacity by distorting the meniscus. In instruments calibrated to deliver the indicated volume, it also causes irregularities by affecting the residue adhering to the walls.

Since accuracy in the volume contained or delivered is possible only when the liquid will form a continuous film on the walls of the measuring vessel, this condition is prescribed as a test of cleanliness.

But even when the surface of the apparatus fulfills this condition, variations in the apparent capacity may still occur, caused by contamination of the liquid surface by minute quantities of fatty or other organic substances. This effect is due to the formation of a film on the surface which produces a change in the surface tension and a consequent variation in the shape of the meniscus.

Various cleaning agents are in use:

1. Fuming sulfuric acid.
2. Chromic-sulfuric acid mixture.
3. Fuming nitric acid.
4. Tri-sodium phosphate.
5. Organic solvents, followed by one of the above agents.
6. Soap and water, followed by one of the above agents.

After cleaning, ware should be well rinsed with tap water and then with distilled water. A final rinse with alcohol is frequently used, following which the piece is dried by evaporation. The process of drying can be hastened, if desired, by blowing *clean, dry* air into the vessel. Efficient scrubbers must be provided to remove any particles of oil or dirt in the compressed air. It has been noted that some alcohols, even though quite pure, contain sufficient wetting agents which remain on the glass as a thin film to lower the surface tension appreciably. Care should be exercised in using such material to hasten the drying.

Never heat a piece of graduated glassware in order to burn out an organic residue.

Method of Setting Liquid on Mark and Reading Meniscus:

Even clean apparatus may give incorrect values unless the meniscus level is set correctly. When using mercury, it will be found necessary to tap the piece sharply several times for the meniscus to assume a shape which can be duplicated.

If the liquid used wets glass, the apparatus should be filled to a point several millimeters above the mark to be tested, and the meniscus gradually lowered to the mark. If it is not practical to use this method, then the container must be shaken slightly, or the inlet tube adding the liquid should be so constructed that the liquid touches the side of the container being tested a short distance above the mark. In this way the glass surface will be wet above the mark and the meniscus will tend to assume the correct shape.

It is general practice to read the top of a mercury meniscus. Care must be taken to shield the top of the mercury from strong light to prevent errors caused by reflection from the surface of the mercury.

Where water or other wetting liquid is used, the method of reading advised by the National Bureau of Standards[4] is as follows:

> "*Method of Reading.*—In all apparatus where the volume is limited by a meniscus the reading or setting is made on the lowest point of the meniscus. In order that the lowest point may be observed it is necessary to place a shade of some dark material immediately below the meniscus, which renders the profile of the meniscus dark and clearly visible against a light background. A convenient device for this purpose is a collar-shaped section of thick black rubber tubing, cut open at one side and of such size as to clasp the tube firmly.
>
> "The position of this lowest point of the meniscus with reference to the graduation line is such that it is in the plane of the middle of the graduation line. This position of the meniscus is obtained by making the setting in the center of the ellipse formed by the graduation line on the front and the back of the graduated tube as observed by having the eye slightly below the plane of the graduation line. The setting is not made at either the top or at the bottom of the line."

If the line to be read is less than a half circle, the same general method should be followed, but the position of the lowest point of the meniscus with respect to the middle of the graduation line must be estimated. Care must be taken to have all pieces to be tested supported firmly with the vertical axis truly perpendicular; otherwise serious errors may be introduced when reading the liquid levels.

Testing Liquids:

The two liquids in general use for calibration are water and mercury. When calibration is performed by weighing the quantity of water or mercury contained or delivered, it is necessary to use pure materials. For tests made by transferring from one vessel into another, clean tap water or ordinary clean, dry mercury may be used. The tap water should not be taken directly from the line, but should be led first into a reservoir to get rid of air bubbles and solid material.

CALIBRATION

Gravimetric Calibration:

Calibration of all apparatus that delivers the contents through a tip should be done by weighing the quantity of water delivered. (Volumetric methods for such items involve complicated setups to allow for drainage volumes.) Flasks may also be tested in this way, although it is usually quicker to use a volumetric method.

To test pipettes or similar instruments ordinarily filled by suction by weighing the water delivered, clamp the instrument in a vertical position, and fill by suction from a beaker of water. A convenient setup is shown in Fig. 1. The pipette is filled by suction applied through the rubber hose at top, emptying the water back into the beaker one or more times to bring the pipette to the temperature of the water. First observing the temperature of the water in the beaker, the pipette is again filled to a short distance above the mark, the stopcock closed and the beaker removed and the outside of the tip wiped with clean filter paper to remove adhering drops. A clean beaker is held touching the tip and the liquid let out slowly by opening the stopcock and controlling the delivery by a finger held on tube *A* until the meniscus descends to the mark. The beaker is removed and the pipette contents then delivered into the weighing flask.

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[4] Communication from National Bureau of Standards.

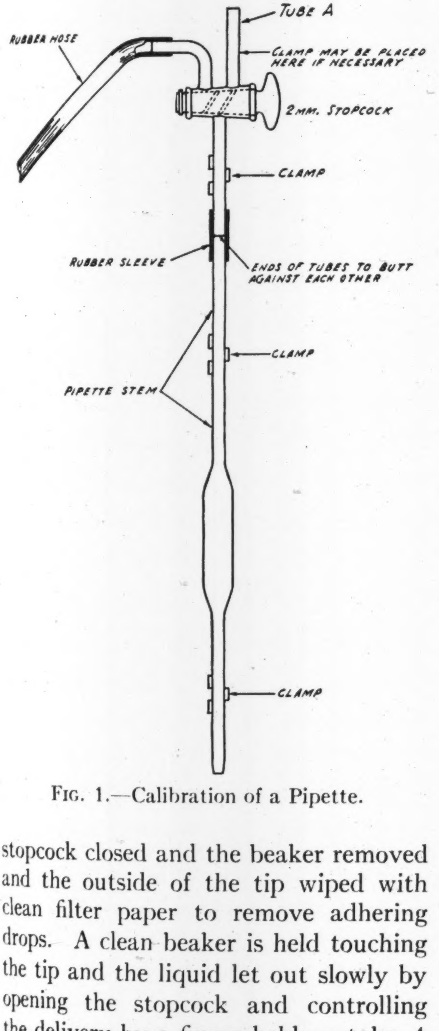
TUBE A
RUBBER HOSE
CLAMP MAY BE PLACED HERE IF NECESSARY
2 MM. STOPCOCK
CLAMP
RUBBER SLEEVE
ENDS OF TUBES TO BUTT AGAINST EACH OTHER
CLAMP
PIPETTE STEM
CLAMP

FIG. 1.—Calibration of a Pipette.

Burettes are handled in about the same way, except that filling is done from the top.

Burettes and measuring pipettes are *always* to be tested in intervals beginning with the zero mark.

To obtain the apparent weight in air of the water contained by a flask, the latter is weighed empty and again after being filled with water to the mark. A stopper should be used to lessen evaporation.

The method of weighing by substitution should always be employed to eliminate the effect of any inequalities in the balance arms.[5] The flask to be calibrated or the weighing flask when pipettes and burettes are being calibrated is hung from one balance hook, or supported on a saddle placed on the pan. On the opposite pan is placed a weight heavier than the filled flask. Sufficient weights are placed on the pan with the flask to secure equilibrium. When the flask is filled, weights are removed from this pan. The difference in the amount of weights necessary to secure balance is the apparent weight of the water.

The volume of the water at 20 C. can be determined from the apparent weight in air by using tables prepared by the National Bureau of Standards,[6] when the instruments are made of glasses having a coefficient of cubical expansion near 0.000025 per deg. Cent. Similar tables for other glasses may be constructed by use of the following formula:

$$C = V\left[1 - \frac{[1 + a(t - 20)](\rho - \sigma)\Delta}{\Delta - \sigma}\right]$$

where:

C = correction to be applied,

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[5] C. A. Briggs and E. D. Gordon, "Weighing by Substitution," *Technologic Paper No. 208*, Nat. Bureau Standards (1922).

[6] Anonymous, "Standard Density and Volumetric Tables," *Circular No. 19*, Sixth Edition, Tables 40 to 52, incl., Nat. Bureau Standards (1924).

V = capacity in milliliters at 20 C.,

a = coefficient of cubical expansion of the glass in milliliters per milliliter per degree Centigrade,

t = temperature in degrees Centigrade of the water weighed,

ρ = density in grams per milliliter of water at t,

σ = density in grams per milliliter of the air at the time of weighing, and

Δ = density in grams per milliliter of the weights used.

A full discussion of the construction of such tables can be found in a book by Verney Stott.[7] The effect of buoyancy of the air on weighings is described in a publication of the Bureau of Standards.[8]

While volumetric apparatus is usually calibrated for distilled water, burettes for gas analysis are sometimes specified to be graduated for mercury. Values of the apparent weight of mercury at different temperatures can be found in Smithsonian Physical Tables.[9]

A good balance must be used, and the weights should be equal in accuracy to the class S weights listed in various apparatus dealers' catalogs. The thermometer should be subdivided to 0.1 C., and preferably certified by the National Bureau of Standards. Where the temperature correction is large enough to affect appreciably the results, the thermometer corrections should be applied. The temperature of the testing liquid can be taken either from the reservoir used in filling the object to be weighed, or from the vessel into which the liquid is transferred for weighing.

Volumetric Calibration:

Methods based on transfer of water from a standard container into the instrument to be tested can be utilized for items such as flasks, cylinders, and centrifuge tubes. It will be found necessary to clean all pieces before testing and also to clean the standards frequently during use.

For flasks and similar items either the well-known Morse burettes or vacuum standards similar to those developed at the National Bureau of Standards are suitable (see Fig. 2). The vacuum standards are calibrated gravimetrically to deliver slightly less than the nominal volume desired, and the balance of the capacity is added from a fine-bore burette.

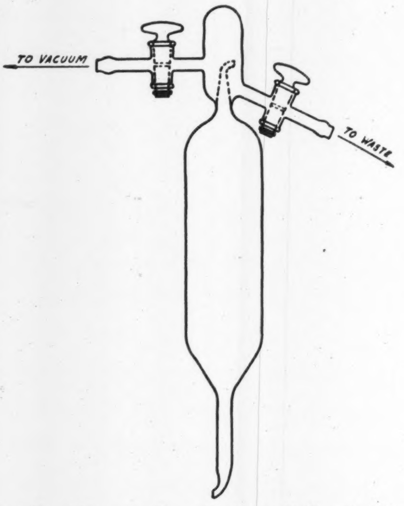

FIG. 2.—Vacuum Standard for Calibration of Flasks.

Small cylinders and centrifuge tubes are best checked by means of a burette. In selecting the burette to use, consideration must be given to the accuracy desired in the calibration and a burette used capable of being read to this degree. For example, in testing a 100-ml. tapered centrifuge tube, a 50-ml. or 100-

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[7] Verney Stott, "Volumetric Glassware," pp. 18-37 Witherby, London. (1928).

[8] Anonymous, "Design and Test of Standards of Mass," *Circular No. 3*, Third Edition, pp. 53-65, Nat. Bureau Standards (1918).

[9] F. E. Fowle, "Smithsonian Physical Tables," Seventh Revised Edition, p. 121 (1923).

ml. burette is satisfactory for the upper part of the scale, but the first few milliliters should be checked by a burette of about 5-ml. capacity subdivided to 0.01 ml.

Another precaution to be taken is to have the delivery stem of the burette long enough to extend almost to the surface of the water in the piece being tested to avoid splashing.

For routine checking, it is sometimes desirable to use mercury, transferring the desired volume from a standard into the piece to be tested. Due to the fact that the volume and shape of the meniscus differs for different liquids, this method will work only when the standard itself has been calibrated with the liquid which has been specified for calibration of the pieces being tested. For the same reason the standard should have the same diameter as the piece at the calibration mark. Checking a shipment in this manner will save considerable time in picking out obviously inaccurate pieces, but this method should not be used as a basis for rejecting doubtful pieces. The latter must always be rechecked by one of the more accurate methods outlined previously.

REPORT OF COMMITTEE E-2
ON
SPECTROGRAPHIC ANALYSIS

Committee E-2 on Spectrographic Analysis held one meeting during the year at Atlantic City, N. J., on June 26, 1940.

The committee sponsored a Symposium on Spectrographic Analysis during the 1940 annual meeting of the Society at Atlantic City, N. J. The symposium, held in conjunction with a Symposium on Tools of Analytical Chemistry, discussed the accuracy of spectrographic analysis in relation to engineering requirements. The discussion papers presented on this occasion were printed in the ASTM BULLETIN, January and March, 1941, and are available as a group in reprint form.

The committee in 1938 sponsored the publication of the "Index to the Literature on Spectrochemical Analysis, 1920–1937," compiled by W. F. Meggers and B. F. Scribner. A revised edition of this bibliography covering the years 1920 to 1939 was published by the Society in April of this year.

One new member was added to the committee and one member lost by resignation, leaving the total membership of 60 members unchanged.

Subcommittee II on Fundamental Methods and Technique (O. S. Duffendack, chairman) is preparing a statement of a specific example of the procedure for the quantitative spectrochemical analysis of a solution by the method of internal control. This will be in a form such that it can be compared with a statement of the procedure of analysis by the method of direct comparison with standards such as that of the A.S.T.M. Tentative Method of Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 40 T) on the analysis of zinc-base alloy die castings.

Subcommittee III on Quantitative Methods and Applications (M. L. Fuller, chairman) held a meeting at Atlantic City on June 26, 1940. In accordance with the action taken at this meeting, the membership lists of the subcommittee and its several groups were reduced in size in the interests of more efficient committeee activity.

Subsequent to the 1940 annual meeting, on the recommendation of this subcommittee, Committee E-2 presented to the Society through Committee E-10 on Standards a revision of the Tentative Method of Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 – 40 T). This recommendation was accepted[1] by Committee E-10 on November 5, 1940, and the revised tentative method has been published separately as a 1940 Supplement to the Methods of Chemical Analysis of Metals.

Groups 1, 2, 4, and 5 also held meetings at Atlantic City on June 26, 1940.

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[1] In submitting this recommendation to Committee E-10 on Standards, Committee E-2 reported results of the letter ballot vote as follows: Of a total membership of 62, 37 members returned their ballots, of whom 24 voted affirmatively, 0 negatively, and 13 marked their ballots "not voting."

Group 1 on copper, nickel and their alloys (P. A. Leichtle, chairman) has been especially active. A standard brass sample is being analyzed by the members to compare existing methods of analysis; outlines of the methods are being prepared.

Group 2 on lead, antimony, bismuth and their alloys (A. E. Ruehle, chairman) is preparing methods for the analysis of tin metal, solders, and tin-base die-cast alloys.

Group 4 on zinc, cadmium and their alloys is engaged in a cooperative test among its members employing the method for the analysis of a zinc die casting alloy, Tentative Method E 27 – 40 T. Experimental work is in progress on an internal standard method for the analysis of zinc-base die-casting alloys. A method for the analysis of cadmium is under consideration.

Subcommittee V on Standards and Pure Materials (T. A. Wright, chairman) has announced that nickel of high purity is available in limited amounts for use in spectrographic work. This subcommittee has supplied information with respect to various pure materials in answer to inquiries.

This report has been submitted to letter ballot of the committee which consists of 60 members; 44 members returned their ballots, of whom 40 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. V. CHURCHILL,
Chairman.

B. F. SCRIBNER,
Secretary.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS

Committee E-3 on Chemical Analysis of Metals held a meeting in Atlantic City, N. J., on June 25, 1940, during the annual meeting of the Society. Four division and nine subcommittee meetings were also held during the year. At the 1940 annual meeting in Atlantic City the committee sponsored a Symposium on Tools of Analytical Chemistry. This symposium was held in conjunction with a Symposium on Spectrographic Analysis, at both of which the A.S.T.M. acted as host to members of the Analytical Division of the American Chemical Society. The papers presented on this occasion were printed in the ASTM BULLETIN.[1]

Four new members were elected to the committee during the year, and one resignation was received. The committee wishes to record its sorrow in the loss by death of Alfred M. Smoot, long an esteemed and valued member of analytical committees of the A.S.T.M.

Division A on Ferrous Metals (Arba Thomas, chairman) has prepared methods for the determination of aluminum, lead, and columbium in steel that have been approved by Committee E-3 by letter ballot and will be submitted to the Society through Committee E-10 on Standards later in the year[2] for publication as tentative to be added, when adopted, to the Standard Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 39). Division A is preparing methods for the determination of aluminum in alloy steels, small amounts of lead in carbon steels, and selenium in stainless steels.

Subcommittee A-2 on Metallic Materials for Electrical Heating (T. R. Cunningham, chairman) has completed the preparation of methods for the analysis of electrical heating alloys. These methods have been approved by letter ballot by Division A and will be submitted to letter ballot of Committee E-3.

Division B on Non-Ferrous Metals (R. P. Nevers, chairman) through its subcommittees is continuing the complex and extensive task of revising the present A.S.T.M. methods for the analysis of non-ferrous alloys. Splendid progress has been made in this work during the past year.

Subcommittee B-1 on Copper and Nickel and Their Alloys (R. P. Nevers, chairman) has completed the preparation of methods for the analysis of brasses. These include procedures for the determination of copper, zinc, lead, tin, iron, phosphorus, arsenic, antimony, and nickel. These methods are now being tested on two samples of brass which represent commercial compositions.

Subcommittee B-2 on Lead, Tin, Antimony, Bismuth and Their Alloys (J. B. Mosley, chairman) has prepared methods for the analysis of pig lead that have been approved by letter ballot by Division B and will be submitted to letter ballot of Committee E-3. If approved, these methods will then be presented to the Society to replace the present Tentative Methods of Chemical Analysis of Pig Lead (B 35 - 36 T). Subcommittee B-2 has undertaken a revision of the Tentative Methods of Chemical Analysis of Alloys of Lead, Tin, Antimony, and Copper (B 18 - 36 T). In connection with this work a special sample of solder (35 per cent tin, 65 per cent lead) has been prepared for analysis.

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[1] ASTM BULLETIN, No. 108, January, 1941, p. 21; also available as a separate publication.
[2] See Editorial Note, p. 501.

Subcommittee B-3 on Aluminum and Magnesium and Their Alloys (H. V. Churchill, chairman) has prepared a revision of the Tentative Methods of Chemical Analysis of Aluminum and Light Aluminum Alloys (B 40 - 36 T). These revised methods are being tested by members of the subcommittee on two samples of aluminum-base alloys specially prepared for this work. This subcommittee has also drawn up methods for the analysis of magnesium alloys, and samples of these alloys are being prepared for use in a cooperative study of these methods by the subcommittee.

Subcommittee B-4 on Zinc and Its Alloy (R. W. Coltman, chairman) has prepared methods for the analysis of slab zinc and submitted them to letter ballot in Division B. This subcommittee is now working on methods for the analysis of zinc-base alloys.

Division D on General Analytical Methods (B. L. Clarke, chairman) has reviewed methods for the analysis of slab zinc, zinc-base alloys, nickel, and nickel-chromium electrical heating alloys that have been submitted by Divisions A and B.

This report has been submitted to letter ballot of the committee which consists of 67 members; 65 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL,
Chairman.

H. A. BRIGHT,
Secretary.

EDITORIAL NOTE

Subsequent to the annual meeting Committee E-3 presented to the Society through Committee E-10 on Standards a tentative revision of the Standard Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 - 39). This tentative revision in the form of new Tentative Methods of Chemical Analysis for Aluminum, Columbium, and Lead in Steel (E 30 - 41 T), was accepted by Committee E-10 on October 22, 1941, and has been published separately as a 1941 Supplement to the Methods of Chemical Analysis of Metals.

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY

Committee E-4 on Metallography held a meeting during the A.S.T.M. spring committee meetings in Washington, D. C., on March 6, 1941, at which time the subcommittee activities were reviewed and plans made for the increase in scope of some of the subcommittees.

The membership of Committee E-4 shows a further increase and now numbers a total of 67 members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (G. F. Comstock, chairman).—There has been need for but very little activity in this subcommittee during this year as the result of having completed the current Tentative Methods of Preparation of Metallographic Specimens (E 3 – 39 T). The methods are being continued as tentative without revision.

Subcommittee IV on Photography (L. V. Foster, chairman).—Because of the apparent advisability of revising the present non-ferrous grain size counting methods included in the Tentative Methods of Preparation of Micrographs of Metals and Alloys, Including Recommended Practice for Photography as Applied to Metallography (E 2 – 39 T),[1] it has been decided to remove Sections 7 to 11, inclusive, and Plate III (grain size chart) from the methods and place these items under the jurisdiction of Subcommittee VIII. Subcommittee VIII will use this material as the basis for a separate standard for determining the grain size of non-ferrous metals, as mentioned in detail later in this report. Pending completion of the proposed revisions, Methods E 2 – 39 T are to be continued as tentative in their present form.

Subcommittee VI on X-Ray Diffraction (W. L. Fink, chairman).—This subcommittee is now preparing recommended practices for five of the more important procedures common to the metallographic applications of X-ray diffraction and plans to have this work completed within the year.

The efforts of this subcommittee, working in conjunction with the National Research Council Committee on X-Ray and Electron Diffraction in order to make available for general use the method of chemical analysis by X-ray diffraction, have now reached the stage where the file catalogs of materials already identified will soon be obtainable.

Subcommittee VII on Dilatometry (L. H. Carr, chairman).—This subcommittee is preparing to correlate the information obtained to date in their survey of dilatometric methods and hopes to have these in such form as to permit the formulation of some recommended practices in the near future.

Subcommittee VIII on Grain Size (M. A. Grossmann, chairman).—Because of the necessities of additional work on grain count methods, the activities of this subcommittee are being enlarged to permit the formation of an additional subgroup devoted to the study of ferrite grain size. Another subgroup will prepare those sections dealing with the metallographic grain size chart for nonferrous metals and alloys now appearing in the Tentative Methods E 2 – 39 T in the form of a separate standard. This will not only constitute some revision of the present method but will also augment present procedure by the use of the grain-size comparator such as described in the ASTM BULLETIN.[2]

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[1] 1939 Book of A.S.T.M. Standards, Part I, p. 1214.

Subcommittee IX on Inclusions (Samuel Epstein, chairman).—Intensive work by this recently formed subcommittee is progressing in a very satisfactory manner. The subcommittee has completed a survey of the various methods of measurement of nonmetallic inclusions, and is currently making a practical comparison of all known methods by having the subcommittee members rate the same kinds of steels by these methods. This procedure should bring out the desirable and unsatisfactory features in each method and thus provide a basis for an improved procedure.

Committee E-4 has reviewed and approved the proposed Tentative Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron[3] prepared jointly by Committee A-3 on Cast Iron in cooperation with the Gray Iron Division of the American Foundrymen's Association.

This report has been submitted to letter ballot of the committee, which consists of 65 members; 35 members returned their ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

J. J. BOWMAN,
Secretary.

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[2] Published in ASTM BULLETIN, No. 111, August, 1941, p. 45.

[3] 1941 Supplement to Book of A.S.T.M. Standards, Part I, p. 300.

REPORT OF COMMITTEE E-8

ON

NOMENCLATURE AND DEFINITIONS

A meeting of Committee E-8 on Nomenclature and Definitions was held during the annual meeting of the Society in Chicago, Ill., on June 25, 1941. At this meeting consideration was given to a number of recommendations for the adoption as standard of certain tentative definitions appearing in the current reports of several standing committees of the Society, and to other matters dealing with nomenclature and definitions of general interest to the committees of the Society.

Under the rotational basis on which the Advisory Committee is now functioning with the terms of two members expiring each year, the Executive Committee of the Society has appointed Messrs. M. E. Holmes and G. B. Waterhouse for a three-year term, expiring in 1943.

The new definitions and revisions in existing nomenclature that have been prepared by the standing committees of the Society give evidence of an increasing interest in the establishment of authoritative nomenclature and definitions. This is especially true in the case of new committees organized during the past several years.

The Joint Committee on Definitions of Terms Relating to Heat Treatment, responsible for the preparation of the Standard Definitions of Terms Relating to Heat Treatment Operations (Especially as Related to Ferrous Alloys) (A 119 – 33), was recently reorganized to give consideration to revision of these definitions and to make whatever extension in the list that seems to be desirable, possibly including the use of heat treatment definitions as applied to non-ferrous metals and alloys. This joint committee is composed of representatives of the three sponsoring societies, namely, the American Society for Metals, the Society of Automotive Engineers, and the American Society for Testing Materials. The definitions should also be of interest to the American Foundrymen's Association and that organization may appoint an official representative to serve on the joint committee. Since several of the A.S.T.M. standing committees are concerned with nomenclature relating to heat treatment, an E-8 subcommittee on this subject has been organized which will cooperate with the joint committee. The personnel of this subcommittee is as follows:

Lawford H. Fry (chairman) Representing Committee A-1 on Steel,
P. V. Faragher,
John Howe Hall,
J. S. Vanick, Representing Committee A-3 on Cast Iron,
B. H. DeLong, Representing Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys,
Alfred Bellis, Representing Committee B-1 on Copper and Copper-Alloy Wires for Electrical Conductors,
Carter S. Cole, Representing Committee B-5 on Copper and Copper Alloys,
G. O. Hiers, Representing Committee B-6 on Die-Cast Metals and Alloys, and
H. J. Rowe, Representing Committee B-7 on Light Metals and Alloys.

Mr. Fry is also one of the A.S.T.M. representatives on the joint committee. The Society's other representatives on the joint committee are T. S. Fuller and H. S. Rawdon.

Committee D-5 recommended[1] to the Society at this annual meeting the withdrawal of the Tentative Definition of the Term Coke (D 121 – 26 T) and requested Committee E-8 to take the initiative in formulating a definition of this term. The committee voted not to attempt to define the term coke at this time.

The committee has reviewed and approved for adoption as standard the revised definition of water-gas tar recommended by Committee D-7 on Timber. This definition is also of interest to Committees D-8 on Bituminous Waterproofing and Roofing Materials and D-4 on Road and Paving Materials, and this action was subject to approval of the definition by the Subcommittee on Definitions Relating to Bituminous Materials (K. G. MacKenzie, chairman). The results of the vote in Committee E-8 on this recommendation are as follows: From a membership of 52, 39 members returned their ballots, 18 of whom voted affirmatively, 0 negatively, and 21 members marked their ballots "not voting."

A revision of the Tentative Definition of the Term Aggregate (C 58 – 28 T) has been prepared and will be reviewed by the subcommittee on this subject.

The following actions were taken by the committee on tentative definitions being recommended for adoption as standard by other Society committees at this annual meeting:

The committee approved the five revised definitions relating to magnetic testing proposed by Committee A-6 on Magnetic Properties for inclusion in the Standard Definitions of Terms with Units and Symbols Relating to Magnetic Testing (A 127 – 39).

The eleven definitions relating to textile materials published as tentative under the designation D 123 – 40 T, recommended for adoption as standard by Committee D-13 on Textile Materials, were reviewed by Committee E-8. Attention was called to the fact that several of these definitions are of a general nature and accordingly are of interest in connection with the work of other standing committees. In view of this, it was considered desirable to defer adoption as standard of these definitions so that there will be opportunity to discuss with Committee D-13 certain desirable improvements in the definitions.

The committee also reviewed the definitions of "abrasion," "corrosion," "erosion," and "slagging" appearing in the Tentative Definitions of Terms Relating to Refractories (C 71 – 40 T) being recommended for adoption as standard by Committee C-8 on Refractories. These terms, likewise being of a general nature, are of interest to other standing committees and attention was called at the meeting to several changes that appear to be desirable. Action was accordingly taken to request that the definitions be not adopted as standard at this time in order that suggested modifications may be discussed with Committee C-8.

The adoption as standard of the revised definitions of the terms gypsum lath, gypsum sheathing board, and gypsum wall board, prepared by Committee C-11 on Gypsum to replace the definitions appearing in the Tentative Definitions of Terms Relating to Gypsum (C 11 – 40 T) was approved for reference to letter ballot in Committee E-8. The results of the vote on this recommendation are as follows: From a membership of 52, 38 members returned their ballots, 21 of whom voted affirmatively, 1 negatively, and 16 members marked their ballots "not voting."

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[1] See p. 372.

The recommendation for adoption as standard of the Tentative Definition of the Term Screen (Sieve) (E 13 - 28 T), with a revision in Note 2 changing "rectangular" to read "square," was approved by letter ballot of the Subcommittee on Sieve and Screen (Cloyd M. Chapman, chairman). Action was accordingly taken to refer this definition to letter ballot in Committee E-8 for presentation to the Society in 1942 for adoption as standard.

At its meeting the committee discussed briefly a number of new and revised definitions prepared by six of the standing committees. Action was taken to refer these by letter to members of the committee for further study and comment.

A new Subcommittee on Definition of Glass is to be organized to reconcile the differences between the definition as prepared by Committee C-14 on Glass and Glass Products and the existing definition for the term glass prepared by Committee D-13 on Textile Materials.

In connection with its work, the committee has recognized a need for a statement of guiding principles to be used in developing definitions and standard forms for expressing them. It is believed this will be of distinct value to the standing committees of the Society. An article along these lines is to be prepared for early publication in the ASTM BULLETIN for the purpose of eliciting discussion on this subject. The article will be based on statements that have been prepared by H. C. Porter and J. S. McDowell.

This report has been submitted to letter ballot of the committee which consists of 52 members; 39 members returned their ballots, of whom 37 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

CLOYD M. CHAPMAN
Chairman.

R. E. HESS,
Ex-officio Secretary.

REPORT OF COMMITTEE E-9

ON

RESEARCH

In the following there is presented a brief résumé of certain principal research activities during the past year which have engaged the attention of Committee E-9 on Research, with particular reference to new projects and cooperative research. A more comprehensive review of research activities throughout the Society will appear in the December issue of the ASTM BULLETIN.

Research Committee Activities:

The *Research Committee on Fatigue of Metals* is presenting to the Society at this annual meeting its final report on the investigation of effect of type of testing machine on results of fatigue tests,[1] towards the expense of which $700 was provided from the Research Fund. This report presents a detailed description of and results obtained in the investigation of the effect of different types of testing machines on the endurance limit of metals and on the shape of the *S-N* (stress-cycle) diagram. The machines studied included the rotating beam, the rotating cantilever beam, vibratory flexural, and direct axial stress. The tests were made on high-strength heat-treated alloy steel, medium-strength low-carbon steel, and an aluminum alloy (duralumin). The report presents profilograph records, magnaflux tests, and photomicrographs of the metals studied, together with a series of curves and a discussion of the variation of endurance limits obtained on the different machines; also the effect of shape of specimen and the endurance above the endurance limit.

The Research Committee is now planning an investigation of effect of size of specimen on the limiting fatigue strength, or endurance limit, of some metals. The general effect of size has been known for some time but its magnitude even for small test specimens is surprising. The committee will include a study of size effect on notched specimens, as compared with size effect for specimens free from notches, and, if found feasible, a study of the effect of residual stresses on fatigue strength. Committee E-9 has appropriated the sum of $500 from the Society's Research Fund in support of this program.

Committee E-9 has requested the Research Committee to consider the inauguration of more extensive researches in the field of fatigue of metals, including problems that might be raised in connection with the utilization of materials in the defense program. Industrial support for such researches is invited. In this connection the work of the Research Committee on Fatigue of Metals has been brought to the attention of the National Defense Research Committee and its services offered wherever they may be of assistance.

The *Joint Research Committee on Effect of Temperature on the Properties of Metals* of The American Society of Mechanical Engineers and this Society is presenting a report[2] at this annual meeting of the Society briefly summarizing the progress made during the year in the various research projects sponsored by the joint committee, including tests of tubular members at elevated temperatures, properties of metals at low temperatures, effect of variables on the high-temperature properties of metals, relaxation tests, and study of high-temperature test methods.

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[1] See p. 133.

[2] See p. 154.

A report on Impact Resistance and Tensile Properties of Metals at Subatmospheric Temperatures,[3] prepared by H. W. Gillett for Project No. 13 of the Joint Research Committee, has also been completed this year. The information contained in this report, in the convenient form provided, is expected to be particularly valuable at this time to the War and Navy Departments and it is believed the National Defense Research Committee will also be interested in the availability of this report.

The *Joint Research Committee on Boiler Feedwater Studies* has continued its research on study of the problem of caustic embrittlement. This study has had many ramifications including (*a*) the development of several types of apparatus by which it would be possible to determine if a given boiler water would cause metallic specimens to crack, (*b*) a conscientious effort to correlate with the findings of the Bureau of Mines' study the work of the University of Illinois, which in all phases did not agree with the results of the committee's research, and (*c*) the critical examination and testing of several preventive chemical reagents. A progress report[4] describing the activities of the committee during the year is to be presented at this annual meeting.

The *Joint Committee on Exposure Tests of Plating on Non-Ferrous Metals*, formerly under the auspices of the National Bureau of Standards, American Electroplaters' Society, and A.S.T.M., has now been discharged since its activities will be carried on in the newly organized Committee B-8 on Electrodeposited Metallic Coatings.

Joint Research Project on Durability of Concrete.—As mentioned in the report of Committee E-9 last year, the governing boards of the Highway Research Board of the National Research Council, the American Concrete Institute, and A.S.T.M., realizing the need that exists for basic research on the fundamental factors that affect the durability of portland-cement concrete, agreed to organize a joint research project on this subject. This research project has now been inaugurated through the appointment of an Administrative Committee, consisting of two representatives from the three sponsoring bodies, and a Technical Committee (appointed by the Administrative Committee) to prepare and supervise the work program. A. T. Goldbeck and the Secretary-Treasurer represent the Society on the Administrative Committee. A full account of the organization of the project and the personnel of the Administrative and Technical Committees appeared in the ASTM BULLETIN, No. 108, January, 1941, p. 52.

Research Progress of A.S.T.M. Committees:

Determination of Water Vapor in Gaseous Fuels (Committee D-3).—The investigation on this subject being carried out for Committee D-3 at The Pennsylvania State College under the direction of Prof. A. W. Gauger is described in a paper by F. C. Todd and A. W. Gauger on "Studies on the Measurement of Water Vapor in Gases"[5] on the program for this annual meeting. The grant of $200 from the Research Fund made in support of this investigation has been used to defray the cost of a voltage-regulating transformer, a centrifugal circulation pump and associated equipment required to complete the tests.

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[3] Issued as a separate publication, August, 1941.

[4] Published in ASTM BULLETIN, No. 111, August, 1941, p. 56.

[5] See p. 1134.

Capping Tests of Hollow Tile (Committee C-15).—A report on this project carried out at Rensselaer Polytechnic Institute for Committee C-15 on Manufactured Masonry Units has been submitted to the committee.

Study to Determine if Results of Empirical Flow Tests can be Expressed in Absolute Units (Committee E-1).—Committee E-9 has upon the request of Committee E-1 on Methods of Testing appropriated $500 from the A.S.T.M. Research Fund for preliminary study to determine if results of empirical flow tests (in particular the ring-and-ball softening point method) can be expressed in absolute units. Arrangements have been made for the investigation to be carried out at Brooklyn Polytechnic Institute under the direction of W. H. Gardner and H. Mark, and work has already been started on the committee's program.

Moisture Content of Paperboard and of Corrugated Fiberboard Containers (Committee D-6).—As mentioned in the report a year ago,[6] the work conducted at six laboratories under the direction of Subcommittee IV of Committee D-6 on Paper and Paper Products has been completed and although a report on the actual investigation will probably not be published as such, the data obtained have been useful in the committee's standardization work.

Testing of Textile Finishes (Committee D-13).—The work of the special committee of Committee D-13 on Textile Materials studying this subject received sufficient financial backing from industry to warrant its continuance for a second year. The current phase of the program has been the development of instruments for the evaluation of the "hand" of fabrics.[7] The subscribers to the project have appointed a committee to set up a program for a third year's work and to determine whether financial backing can be secured for it. Another committee has as its function, first, to present to Committee D-13 for adoption instruments and methods which have been developed; second, to promote their general use and to answer inquiries which may arise regarding them; and third, to renew the research program at such time as may be propitious.

Effect of Pretreatments in Painting of Structural Steel (Committee D-1).—This project, under the direction of Subcommittee XXIX of Committee D-1 on Paint, Varnish, Lacquer, and Related Products, has progressed during the year. Paints on the committee's test panels have now been exposed 21 months and several field inspections have been made. A report on the present status of this project will be made by the subcommittee to Committee D-1 this June.[8]

Comparative Fire Tests of Treated and Untreated Wood (Committee C-5).—This project has for its objective the development of a standard method of testing wood chemically treated by impregnation to determine its fire-resistance properties. It comprised studies on five currently used fire test methods applied to treated and untreated wood in which five laboratories cooperated, working independently, and it is considered to be the most extensive series of fire tests of this kind ever made. A complete report of the investigation is included in this year's report of Committee C-5 on Fire Tests of Materials and Construction,[9] which proposes Tentative Method of Test for Fire-Retardant Properties of Wood[10] based on this investigation.

Corrosion Studies.—Probably the largest single research project being sponsored by the Society is the Wire Test Program of Committee A-5 on Corrosion of Iron and Steel. The committee is presenting this year another report,[11] after a two-year interval, covering the results of the atmospheric corrosion tests on wire and wire product specimens after exposure for about four years at each of eleven locations.

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 456 (1940).

[7] See p. 448.

[8] See p. 322.

[9] See p. 238.

[10] 1941 Supplement to Book of A.S.T.M. Standards, Part II, p. 281.

The data obtained in the atmospheric corrosion studies carried out by Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys on some 24 non-ferrous metals and alloys exposed at ten test sites, has been further studied and the current report[12] of the committee presents a statistical analysis of the test data.

New Activities.—In addition to the projects just described, research activities on the following subjects have been started during the year by the committees named. Brief statements of each appeared in the ASTM BULLETIN for October, 1940:

Laboratory Total Immersion and Alternate Immersion Tests on Non-Ferrous Metals (Committee B-3).

Glass Fiber and Its Products (Committee D-13).

Organization of A.S.T.M. Research Activities on Corrosion:

Committee E-9 on Research has been giving some consideration to the status of the Society's researches in the field of corrosion, which have been carried on in at least five committees, namely, A-5 on Corrosion of Iron and Steel, A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, B-3 on Corrosion of Non-Ferrous Metals and Alloys, B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys, and B-6 on Die-Cast Metals and Alloys; and presumably the question of corrosion research regarding electrodeposited coatings will arise in the newly organized Committee B-8 on Electrodeposited Metallic Coatings. As the work has grown throughout the years in volume and importance there has developed to a certain extent what might appear to be duplication of effort and certainly there has developed a need for coordination of this work that goes beyond the possibilities, apparently, of the Coordinating Committee on Corrosion that was set up some years ago. Arrangements have accordingly been made for a conference at this annual meeting under the auspices of Committee E-9 of the advisory committees of all the committees concerned, to consider the possibility of creating in the Society some administrative agency that would direct this important side of its research activities.

Proposed Foundation for Scientific Research in Rubber Technology:

A joint committee of the Society of Automotive Engineers and this Society, which functions as Technical Committee A on Automotive Rubber of A.S.T.M. Committee D-11 on Rubber Products, has been active in the study of certain methods of test for automotive rubber and the relationship of such methods to purchase specification requirements. As the result of these studies over a period of a year, the joint committee is convinced that the method of approach is not an adequate one considering the magnitude of the problem, and has drawn up for consideration by S.A.E. and A.S.T.M. an outline for the formation of a Foundation for Scientific Research in Rubber Technology to determine and obtain scientific answers to technological rubber problems.

Both Committees D-11 and E-9 have considered the proposal and both agree that the A.S.T.M. is definitely interested and should assist in the establishment of a suitable research project. However, research of the magnitude that is here involved must be approached from a broad viewpoint and should have from the beginning the cooperation of the several technical societies and industrial groups that have a major interest in production and utilization of rubber. Accordingly, Committee E-9 has suggested as one method of approach that there be set up under the aegis of Engineering Foundation a project for Scientific Research in Rubber Technology to be sponsored by such societies and organizations as will be representative of producing, consuming, and fabricating interests; and that a suitable administrative committee in charge of the project be established by the sponsoring bodies with authority to form suitable representative committees to be responsible for specific phases of the general project.

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[11] See p. 101.

[12] See p. 172.

Research Activities of Other Societies:

National Research Council.—The Society has continued its contacts with the Division of Engineering and Industrial Research through its representative, L. M. Currie, and also with the Highway Research Board of that Division through Prévost Hubbard, representative, and F. E. Richart, alternate. Mr. Hubbard's activities during the past year were confined to cooperation with the Department of Design, Committee on Flexible Type Surfaces, and he was co-author of a paper on "Required Thickness of Asphalt Pavements in Relation to Subgrade Support" presented at the annual meeting of the Board in December, 1940.

A.S.M.E. Research Committee on Mechanical Springs.—The Society's representative on this committee, C. T. Edgerton, has maintained his contacts with its activities. While direct research activities have been at a standstill during the past year, pending development of new fatigue testing facilities, arrangements have now been made that will enable the committee to proceed with its program.

Engineering Foundation—Welding Research Committee.— N. L. Mochel has continued to represent the Society on the Industrial Research Division of the Welding Research Committee and close contact is being maintained between the activities of the Division and certain interested A.S.T.M. committees. The investigation on weldability of carbon steels, to which the Society has contributed the sum of $250 from the Research Fund, has been started at Battelle Memorial Institute and a first progress report is expected shortly. A progress report on work at Rensselaer Polytechnic Institute on spot welding has been submitted to the National Advisory Committee for Aeronautics. The fatigue investigation on welded joints carried on at the University of Illinois has been reported in an Experimental Bulletin of that University and covered by progress reports of the committee. A number of other projects are under way and are currently reported in *The Welding Journal* published by the American Welding Society.

The committee has prepared three exhibits of its work for the current annual meeting.

American Coordinating Committee on Corrosion:

During the past year the American Coordinating Committee on Corrosion has prepared a Directory of workers in the field, a first draft of which has been submitted to the sponsoring societies and to each of the workers. It is probable that a revised Directory will be prepared as a result of this first circularization.

The Coordinating Committee is sponsoring jointly with the American Association for the Advancement of Science a four-day symposium on corrosion to be held this summer at Gibson Island, Md.

Relationship of A.S.T.M. Research to National Defense:

In its annual report to the Society the Executive Committee has discussed various aspects of the Society's relation to the National Defense Program. After mentioning the significant relationship of A.S.T.M. standardization activities to national defense, it was stated in substance that research in materials is scarcely less important than standardization itself since research provides the knowledge required for the efficient utilization of materials and the information needed to write useful specifications.

There has been formed as a part of the Defense Program the National Defense Research Committee, to correlate and support systematic research on the mechanism and devices of warfare. Committee E-9 has offered to the N.D.R.C. the cooperation of the Society in whatever way the research facilities of A.S.T.M. could be advantageously used in the Defense Program. Descriptions of research activities at present under way in the committees of the Society have been furnished to the N.D.R.C.

Committee E-9 would stress the importance of using existing agencies for research wherever possible in times of emergency such as these. It is to be remembered, of course, that research sponsored by A.S.T.M. is essentially of a cooperative nature. Projects are planned under committee auspices by various groups having a common interest in acquiring certain information and are carried out in ways that frequently involve the pooling of facilities of several research agencies, such as industry and Government laboratories, research foundations, and universities. Thus the Society brings to bear upon its research problems unusual talent and facilities. It is suggested that this cooperative approach to research might be used advantageously by Government departments and possibly other defense agencies to augment their own laboratory studies, especially in matters involved in the working out of production methods and determining specification limits.

Research Exhibits at Annual Meeting:

Emphasis has been placed on the desirability of research exhibits and as a result of invitations extended to various companies, committees, and governmental agencies, several research exhibits have been arranged in conjunction with the Sixth Exhibit of Testing Apparatus and Related Equipment at this annual meeting.

Administrative Matters:

The term of T. S. Fuller expires with this annual meeting and the appointment of a new member for the term of five years will be made by the Executive Committee of the Society.

The principal of the A.S.T.M. Research Fund is now $29,391.15. Since the report a year ago the principal was augmented by $1300 from the entrance fees for 1940 (in addition to the $200 reported last June) and $600 from current (1941) entrance fees.

The balance of income in the Research Fund on May 31, 1941, was $689.12. This is after payment of $500 each to the Research Committee on Fatigue of Metals and Committee E-1 on Methods of Testing mentioned earlier in this report. The present annual available income from the principal of the Fund is about $950.

Respectfully submitted on behalf of the committee,

G. F. JENKS,
Chairman.

C. L. WARWICK,
Secretary.

REPORT OF COMMITTEE E-10

ON

STANDARDS

Committee E-10 on Standards held one meeting during the year: in Philadelphia, Pa., on August 28, 1940.

At that meeting R. P. Anderson was elected chairman of Committee E-10 to serve until June 1941, at which time his term of office as a member of Committee E-10 expires. The membership on Committee E-10 of J. R. Townsend and J. O. Leech expired last year. The Executive Committee reappointed Mr. Townsend and appointed N. L. Mochel of Westinghouse Electric & Manufacturing Co. to serve as members of Committee E-10 for a three-year term, expiring in 1943.

The Executive Committee and Committee E-10 on Standards have considered a suggestion that the marking clauses in A.S.T.M. specifications should contain a requirement that materials or products covered therein be marked or tagged in some suitable manner to indicate conformity with the specifications. This requirement is to provide identification of the material or product as furnished by the manufacturer in accordance with the requirements of the specifications. The Society is not in any sense certifying or guaranteeing compliance with these requirements; the latter remains as heretofore a matter entirely between the parties to the specification. The standing committees have been requested to review the specifications in their charge and to determine prior to the 1942 annual meeting whether marking requirements in accordance with this policy, if not already provided, shall be included; and if so, to include the necessary revisions in their 1942 reports.

As in previous years, a considerable number of new tentative standards and revisions of existing standards and tentative standards have been submitted to Committee E-10 for approval, as given in detail later in this report. For the most part these were considered at the August meeting of the committee.

Review of A.S.T.M. Standardization Activities

A summary of the new and revised tentative standards submitted at the 1940 annual meeting was included in the August issue of the ASTM Bulletin together with a review of the standardization work of the various committees, with statements concerning new standards in prospect. Much of the information, particularly with respect to standardization projects under development in committees, is furnished by the standing committees subsequent to the annual meeting and takes advantage of the fact that many committees plan their next year's work at committee meetings held during the annual meeting.

At the present writing, it is possible to make only a preliminary estimate in terms of new and revised standards of the volume of standardization work accomplished during the year and reported to the Society at this meeting. The reports of standing committees to be presented at the annual meeting include recommendations to the Society substantially as follows:

New tentative standards	75
Revisions of existing tentative standards	77
Tentative revisions of existing standards	21
Tentative standards recommended for adoption as standard	88
Revisions of existing standards reommended for adoption as standard	88
Standards and tentative standards withdrawn or replaced	22

There are at present 581 standards and 371 tentative standards of the Society, making a total of 952, and if the above recommendations are approved this total will become 1005.

CONSIDERATION OF NEW AND REVISED STANDARDS

Proposed new standards accepted by Committee E-10 and published as tentative during the year are as follows:

Tentative Specifications for:

Corrosion-Resisting Chromium-Nickel Steel Sheet, Strip, and Plate for Fusion-Welded Unfired Pressure Vessels (A 240 - 40 T), submitted by Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys,

Soft Solder Metal (B 32 - 40 T), submitted by Committee B-2 on Non-Ferrous Metals and Alloys,

Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 40 T), submitted by Committee B-5 on Copper and Copper Alloys, Cast and Wrought,

Concrete Sewer Pipe (C 14 - 40 T), submitted by Committee C-13 on Concrete Pipe,

Reinforced Concrete Sewer Pipe (C 75 - 40 T), submitted by Committee C-13,

Reinforced Concrete Culvert Pipe (C 76 - 40 T), submitted by Committee C-13,

Cut-Back Asphalt (Rapid Curing Type) (D 597 - 40 T), submitted by Committee D-4 on Road and Paving Materials,

Cut-Back Asphalt (Medium Curing Type) (D 598 - 40 T), submitted by Committee D-4,

Tentative Methods of:

Flexure Testing of Glass (C 158 - 40 T), submitted by Committee C-14 on Glass and Glass Products,

Test for Physical State of Cure of Vulcanized Rubber (T-50 Test) (D 599 - 40 T), submitted by Committee D-11 on Rubber Products, and

Tentative Classification of:

Fireclay Refractories (C 27 - 40 T), submitted by Committee C-8 on Refractories.

Revisions of the following tentative standards and tentative revisions of standards were accepted by Committee E-10:

Tentative Specifications for:

Seamless Copper Tubes (B 75 - 40 T), submitted by Committee B-5 on Copper and Copper Alloys,

Paving Brick (C 7 - 40 T), submitted by Committee C-15 on Manufactured Masonry Units,

Crushed Stone for Water-Bound Base and Surface Courses (D 489 - 40 T), submitted by Committee D-4 on Road and Paving Materials,

Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469–40 T), submitted by Committee D-11 on Rubber Products,

Tentative Methods of:

Hydrostatic Pressure Test on Glass Containers (C 147 - 40 T), submitted by Committee C-14 on Glass and Glass Products,

Polariscopic Examination of Glass Containers (C 148 - 40 T), submitted by Committee C-14,

Thermal Shock Test on Glass Containers (C 149 - 40 T), submitted by Committee C-14,

Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 - 40 T), submitted by Committee D-4 on Road and Paving Materials,

Test for Compression Set of Vulcanized Rubber (D 395 - 40 T), submitted by Committee D-11 on Rubber Products,

Tension Testing of Vulcanized Rubber (D 412 - 40 T), submitted by Committee D-11,

Testing Rubber Insulated Wire and Cable (D 470 - 40 T), submitted by Committee D-11,

Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 40 T), submitted by Committee D-11,

Testing Cotton Fibers (D 414 - 40 T), submitted by Committee D-13 on Textile Materials,

Test for Index of Refraction of Transparent Organic Plastics (D 542 - 40 T), submitted by Committee D-20 on Plastics,

Test for Dielectric Strength of Electrical

Insulating Materials at Commercial Power Frequencies (D 149 - 40 T), submitted by Committee D-9 on Electrical Insulating Materials,

Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 - 40 T), submitted by Committee D-9,

Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 40 T), submitted by Committee D-9,

Test for Local Thickness of Electrodeposited Coatings on Steel (A 219 - 40 T), submitted by Committee A-5 on Corrosion of Iron and Steel,

Quantitative Spectrochemical Analysis of Zinc Alloy Die Castings for Minor Constituents and Impurities (E 27 - 40 T), submitted by Committee E-2 on Spectrographic Analysis,

Standard Specifications for:

Clay Sewer Pipe (C 13 - 40), submitted by Committee C-4 on Clay Pipe,

Structural Clay Load-Bearing Wall Tile (C 34 - 39), submitted by Committee C-15 on Manufactured Masonry Units,

Structural Clay Non-Load-Bearing Tile (C 56 - 39), submitted by Committee C-15,

Standard Methods of:

Freezing-and-Thawing Test of Brick (C 67 - 39), submitted by Committee C-15,

Sample Preparation for Physical Testing of Rubber Products (D 15 - 39), submitted by Committee D-11 on Rubber Products,

Chemical Analysis of Rubber Products (D 297 - 39), submitted by Committee D-11,

Testing Molded Materials Used for Electrical Insulation (D 48 - 39), submitted by Committee D-9 on Electrical Insulating Materials,

Testing Electrical Porcelain (D 116 - 39), submitted by Committee D-9,

Test for Pyrometric Cone Equivalent of Refractory Materials, (C 24 - 35) submittee C-8 on Refractories,

Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 - 36), submitted by Committee C-8,

Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 - 40), submitted by Committee C-8, and

Panel Test for Resistance to Thermal and Structural Spalling for Super Duty Fireclay Brick (C 122 - 40), submitted by Committee C-8.

The withdrawal of the following standards and tentative standards was approved by Committee E-10:

Standard Specifications for:

Solder Metal (B 32 - 21), recommended by Committee B-2 on Non-Ferrous Metals and Alloys,

Copper-Base Alloys in Ingot Form for Sand Castings (B 30 - 36), recommended by Committee B-5 on Copper and Copper Alloys, Cast and Wrought,

Standard Methods of:

Analysis of Roofing Felt for Fiber Composition (D 272 - 34), recommended by Committee D-8 on Bituminous Waterproofing and Roofing Materials,

Chemical Analysis of Rubber Products (D 297 - 39), recommended by Committee D-11 on Rubber Products,

Standard Definitions of:

Fireclay and Alumina-Diaspore Refractories (C 27 - 39), recommended by Committee C-8 on Refractories, and

Tentative Specifications for:

Iron and Steel Filler Metal (Arc-Welding Electrodes and Gas-Welding Rods) (A 205 - 37 T), recommended by Committee A-1 on Steel.

PROMOTION AND EXPANSION OF STANDARDIZATION ACTIVITIES

As indicated in the report of the Executive Committee, every effort is being made to keep the A.S.T.M. standards abreast of technological developments so as to make them of maximum usefulness in connection with defense needs. All of the standing committees are alive to the need for currently reviewing standards under their jurisdiction.

It may also prove to be desirable for the Society to enter new fields and this matter is also receiving the attention of Committee E-10. However, unless the standards that would be developed would be of definite assistance under present conditions, the committee hesitates to recommend the organization of any new committees in view of the pressure that is now on most technical men in industry.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Standards Submitted Under Existing Standards and Proprietary Procedure.—The existing standard method for submitting standards to the American Standards Association, whereby it is necessary to show that the standard is supported by the necessary consensus of those substantially concerned with its scope and provisions, is particularly appropriate for a large number of A.S.T.M. standards. Under this procedure the following methods have been approved during the year by the A.S.A. as American standard:

Standard Methods of:

Testing and Tolerances for Tubular Sleeving and Braids (D 354 - 36; ASA L13-1941).

The Society has been granted proprietary sponsorship for the revisions of standards approved under the existing standards method and, functioning as proprietary sponsor, has submitted to to the A.S.A. during the year revisions of the following standards, all of which have been approved:

Standard Specifications for:

Carbon-Steel Castings for Valves, Flanges and Fittings for High-Temperature Service (A 95 - 40; G17.1—1940),
Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 - 40; G17.3—1940),
Gypsum Plasters (C 28 - 40; A49.3—1940),
Gypsum Molding Plaster (C 59 - 40; A49.4—1940),
Gypsum Pottery Plaster (C 60 - 40; A49.5-1940),
Mineral Iron Oxide (D 84 - 40; K25-1940),
Chrome Yellow (D 211 - 40; K27—1940),
Reduced Chrome Green (D 213 - 40; K28—1940),
Prussian Blue (D 261 - 40; K29—1940),
Reduced Para Red (D 264 - 40; K31—1940),
Chrome Oxide Green (D 263 - 40; K37—1940),

Standard Methods of:

Laboratory Sampling and Analysis of Coal and Coke (D 271 - 40; K18—1940), and
Sampling Coal for Analysis (D 21 - 40; K46—1940).

On the recommendation of the Society, functioning as proprietary sponsor, approval as American standard of the following specifications has been withdrawn:

Standard Specifications for:

Ultramarine Blue (D 262 - 28; K30—1937)

Standards Submitted Under Sectional Committee Procedure.—Reports were received from several of the sectional committees for which the Society is sponsor, submitting standards to the ASA for approval. These reports were transmitted to that association and the following standards have been approved for the first time or reapproved in revised form during the year as American standard:

On the Recommendation of Sectional Committee B36 on Standardization of Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing:

Specifications for Welded and Seamless Steel Pipe (A 53 - 40; ASA B36.1—1940), and
Specifications for Lap-Welded and Seamless Steel Pipe for High-Temperature Service (A 106 - 40; B36.3—1940).

On the Recommendation of Sectional Committee C59 on Electrical Insulating Materials:

Methods of Testing Electrical Insulating Oils (D 117 - 40; C59.2—1941), and
Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 - 38; C59.10—1941).

On the Recommendation of Sectional Committee H4 on Copper Wire:

Specifications for Hard-Drawn Copper Wire (B 1 - 40; H4.2—1941), and
Specifications for Medium-Hard-Drawn Copper Wire (B 2 - 40; H4.3-1941).

On the Recommendation of Sectional Committee L3 on Specifications for Rubber-Lined Fire Hose:

Specifications for Cotton Rubber-Lined Fire Hose for Public and Private Fire Department Use (D 296 - 35; L3.1—1941)

On the Recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants:

Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 40; Z11.7—1940),
Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 40; Z11.8—1940),

- Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 40; Z11.9—1940),
- Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 40; Z11.10—1940),
- Method of Test for Distillation of Natural Gasoline (D 216 - 40; Z11.11—1940),
- Methods of Analysis of Grease (D 128 - 40; Z11.16—1940),
- Method of Test for Precipitation Number of Lubricating Oils (D 91 - 40; Z11.30—1940),
- Method of Test for Knock Characteristics of Motor Fuels (D 357 - 40; Z11.37—1940),
- Method of Test for Unsulfonated Residue of Plant Spray Oils (D 483 - 40; Z11.41—1940), and
- Specifications for Stoddard Solvent (D 484 - 40; Z11.42—1940).

Approval of the following methods as American tentative standard was withdrawn by ASA on the recommendation of Sectional Committee Z11:

Tentative Methods of:

- Test for Sulfur in Petroleum Oils by Lamp Method (D 90 - 34 T; Z11.38—1935),
- Test for Color of Lubricating Oil and Petroleum by Means of ASTM Union Colorimeter (D 155 - 39 T; Z11.34—1939), and
- Test for Neutralization Number of Petroleum Products and Lubricants (D 188 - 27 T; Z11.12—1928).

Sectional Committee Organized.—The following sectional committee of interest to the A.S.T.M. has recently been organized under the procedure of the American Standards Association. The Society has accordingly appointed a representative thereon and an additional representative may be designated at a later date:

Sectional Committee B52 on Classification of Materials for Tools, Fixtures, and Gages, represented by B. H. DeLong of Carpenter Steel Co.

Sectional Committee A37 on Road and Paving Materials.—Due to the rather large size of this committee (129 members—identical with the personnel of the Society's Committee D-4 on Road and Paving Materials) it has proved to be rather unwieldy of operation and difficult to get complete letter ballots. It has also been difficult to divorce the reviewing function of the committee when acting as a sectional committee from its originating function when serving as a standing committee of the Society. It has accordingly been deemed desirable to reorganize the sectional committee in order to simplify the personnel and to make of it purely a group consisting of duly appointed representatives of the interested organizations. Invitations have been extended to about thirty such organizations and acceptances have already been received from a number of them. It is proposed to effect reorganization during the present annual meeting.

Respectfully submitted on behalf of the committee,

R. P. ANDERSON,
Chairman.

C. L. WARWICK,
Ex-officio Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

THE PURPOSE of the Edgar Marburg Lecture is to have described at the annual meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first secretary of the Society.

NATURAL AND SYNTHETIC RUBBERS

SIXTEENTH EDGAR MARBURG LECTURE[1]

BY HARRY L. FISHER[2]

INTRODUCTION

Rubber received its lowly name a century and a half ago from the simple observation that, in the words of Joseph Priestley, it was "excellently adapted to the purpose of wiping from paper the marks of a black lead-pencil." Since then it has become a material of thousands of uses. Its unique properties kept it for decades as a type of material entirely by itself and obtainable only from natural sources, but the ingenuity of man has recently again accomplished "the impossible" and produced rubber-like materials which not only duplicate the properties of nature's product but even go beyond them. The word rubber is used very loosely. It is the name given to the crude rubber of commerce and also to the vulcanized products made therefrom. In this paper an effort will be made to make its proper meaning evident from the manner of its use.

It is not expected that all the uses of rubber will be enumerated here. Such a plan is not desirable. However, the author will endeavor to outline enough of the chemical and physical properties to enable one to understand how rubber can be used in such a wide variety of applications. Synthetic rubbers will then be discussed in some detail, their properties compared with those of natural rubber, and some of their uses described. It must be remembered that the author is an organic chemist and therefore what he has to say will naturally be colored by his training and experiences in his chosen field.

PART I—NATURAL RUBBER

Crude Rubber; Properties and Production:

Although in the early 1800's crude rubber was used in the manufacture of a number of articles, it caused much trouble and loss because it deteriorates in air and sunlight, becoming soft and sticky at slightly elevated temperatures and hard and unyielding at freezing temperatures. Its widespread adoption was therefore curtailed considerably. Charles Goodyear's famous discovery of vulcanization in 1839 changed all this and opened the doorway to the great commercial development of the rubber industry. This development later was given a tremendous impetus by the requirements of the automobile.

Vulcanization enhances the elastic properties of crude rubber and extends them over a wider range of temperatures. Furthermore, vulcanization of rubber decreases its solubility in common solvents and, what is most important, decreases its plastic flow and correspondingly its permanent set. Another way of stating the differences between crude and vulcanized rubber is that crude rubber is elastic and thermoplastic, whereas vulcanized rubber is even more elastic but nonthermoplastic. Properly vulcanized rubber after having been stretched returns to approximately its original position, and the same piece can be stretched time and again with the same results.

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[1] Read on June 25, 1941, before the Annual Meeting of the American Society for Testing Materials, Chicago, Ill.

[2] Director of Organic Research, U. S. Industrial Chemicals, Inc., and Air Reduction Co., Stamford, Conn.

For many years the total supply of crude rubber was comparatively small, the world's annual production being only 20,000 to 40,000 long tons. The great rubber plantations in the Far East were founded in 1876, and, fortunately for the development of the automobile industry, they were beginning to bear when the first automobiles were being manufactured. Production increased rapidly, and since 1934 it has about equaled or exceeded 1,000,000 tons annually. Of this amount 618,000 tons were imported last year into the United States. In the same year only about 4000 tons of synthetic rubbers were produced in this country. About 70 per cent of all the crude rubber produced goes into tires and tubes, and an increasing amount is being used in other parts of automobiles. Until recently, plantations have been producing only about two thirds of their potential output. Today's price of crude rubber is about 20 cents a pound.

Rubber comes almost entirely from the famous Brazilian rubber tree, *Hevea brasiliensis*. Other trees and also vines and shrubs produce rubber, but the *Hevea* tree has won out in the race for supremacy because of its shape, type of bark, and adaptability to plantation methods. The rubber is found as a suspension of tiny droplets in a milk-like fluid known as latex which is found in all parts of the tree except the wood. By cutting into the bark the latex vessels contained therein are severed and the latex oozes out. Latex contains 30 to 45 per cent of its weight of solids, most of which on coagulation of the latex forms the crude rubber of commerce. Rubber comes on the market chiefly as pale crepe and smoked sheet, depending upon the method used in its final preparation. These crude rubbers consist of 92 to 95 per cent of the rubber hydrocarbon, $(C_5H_8)_x$, which is chiefly responsible for their properties. Rubber also comes on the market as the original latex preserved with ammonia. Sometimes the latex is concentrated, for example, by centrifuging, and the solids may thus be increased to as much as around 66 per cent. Even at this higher concentration the latex is fluid, although viscous.

Milling Crude Rubber:

Crude rubber is very tough and ordinarily must be worked on a mill before the necessary compounding ingredients can be mixed into it. Milling rubber causes it to become soft and plastic, and then all kinds of ingredients—powders, abrasives, rubber substitute, oils, waxes —can be mixed in. In all cases, except with solvents, rubber remains the outer phase. Milling of rubber is accompanied by the action of a very small amount of oxygen, so small in fact, that it is difficult to determine it. In the complete absence of oxygen, no "breakdown" is noticeable. The first sight of broken-down rubber makes one wonder how it can ever be converted into useful products, but vulcanization under the proper conditions performs this apparent miracle. Much breakdown is required for some purposes, especially for the extruding of tubes, but generally the breakdown is not made any greater than necessary for the object in mind, since the greater the breakdown, the lower are the final physical properties. In the manufacture of articles from latex in which the rubber is not broken down, the physical properties are very high. Large products, however, cannot yet be made from latex.

Results of Research:

Knowledge of the chemistry and physics of rubber was meager until after the turn of the century. Again, the increase in the use of rubber for tires and tubes gave the impetus for a deeper study of the properties of rubber. Also, the development at the same time of colloid chemistry was a strong factor in the further study of rubber, since it too is a colloid. It is of interest that a great deal of the research work on rubber has been done in the laboratories of the rubber and chemical industries. The discoveries of organic accelerators, of antioxidants, and of the abrasion resistance of compounds reinforced with carbon black, have been of the utmost importance. Not only do organic accelerators reduce the time of vulcanization and thus allow more rapid turnover of molding equipment, but they also give products that have many desirable properties not obtainable by sulfur alone. Vulcanized rubber slowly deteriorates in air and light. Antioxidants have lengthened its life and have made possible applications, especially in engineering, which were impossible before their use. Besides greatly prolonging its life, they increase its resistance to flexing and add other properties of value in the mixing of rubber compounds and in the fabrication of rubber goods.

Protected Rubber Is Permanent:

If rubber is kept away from air and light it does not deteriorate. Rubber products submerged in water last a long time. In certain German lignite mines the workmen are familiar with rubbery fibers known as "monkey hairs." These are now known to consist of fossil vulcanized rubber and are the remains of rubber originally in the twigs of a type of rubber-bearing shrub now found only in Africa. The surrounding lignite contains a small proportion of sulfur and, in the course of thousands of years, the sulfur migrated and caused the formation of prehistoric vulcanizates.

Compounding Rubber:

Rubber is practically always used in the compounded vulcanized state. Unvulcanized rubber is used only in cements, as crepe rubber soles for shoes, and in a few other minor instances. To make rubber of commercial value, it must be mixed with the proper compounding ingredients and then vulcanized. The choice of the proper ingredients and the proper combination and proportions of ingredients makes possible the infinite variety of uses of rubber products. Besides accelerators and antioxidants, many other substances are incorporated into rubber to give the required properties. Carbon black is used, as already mentioned, as the chief reinforcing agent for resistance to abrasion. It also helps to give high tensile strength, high resistance to tear, and stiffness to a compound. The semi-reinforcing blacks also give high tensile strength, good resistance to tear, and fair resistance, to abrasion, along with softness and pliability. Zinc oxide, magnesium carbonate, blanc fixe, and certain clays act somewhat like the semi-reinforcing blacks, although tensile strength and resistance to tear are generally lower when some of them. especially clays, are used. Inert filler such as whiting, infusorial earth, some clays, barytes, and slate flour are used to give body or weight to cheapen compounds when high tensile strength and resistance to abrasion are not necessary. Oils, waxes, tars, and mineral rubbers (gilsonite, etc.) are used as softening agents.

Transparent Rubber:

A good grade of crude rubber is transparent but ordinary vulcanized rubber is not. By using a low proportion of sulfur and by proper choice of accelerators to avoid the later formation of crystalline masses, and by using a basic magnesium carbonate with a refractive index close to that of rubber, it is possible to produce sheets of well vulcanized rubber that are permanently transparent.

Molding:

Practically all rubber articles are molded. Molding makes it necessary to build up rubber compounds with the proper ingredients in order that the final product will be of the desired quality and dimensions. The work of the compounder who plans the compound for a definite purpose is very important. He it is who outlines what must be mixed into rubber in order that such small objects as plungers for hypodermic needles and such large objects as great airplane tires will be properly vulcanized and found to do the work required of them. A mistake is costly, since once the object is vulcanized it cannot be taken apart and done over again; in other words, vulcanized rubber has never been devulcanized.

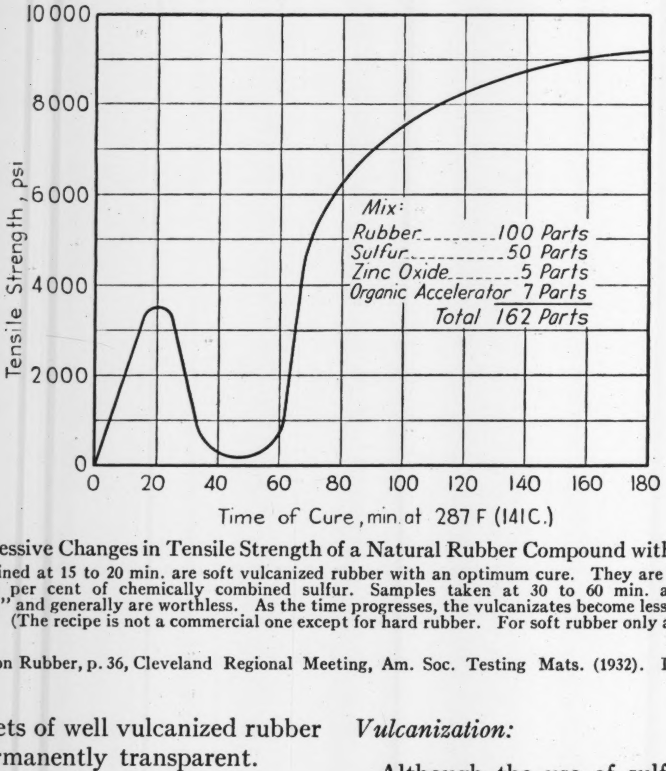


FIG. 1.—Progressive Changes in Tensile Strength of a Natural Rubber Compound with Time of Cure

Samples obtained at 15 to 20 min. are soft vulcanized rubber with an optimum cure. They are highly elastic and contain about 3 per cent of chemically combined sulfur. Samples taken at 30 to 60 min. are still soft, but weak and "short," and generally are worthless. As the time progresses, the vulcanizates become less elastic and finally hard and strong. (The recipe is not a commercial one except for hard rubber. For soft rubber only about 3 per cent of sulfur is used.)

(Symposium on Rubber, p. 36, Cleveland Regional Meeting, Am. Soc. Testing Mats. (1932). Issued as separate publication.)

Vulcanization:

Although the use of sulfur as a vulcanizing agent was discovered over a hundred years ago, it is still practically the only vulcanizing agent used. Other agents are known, but their use is very limited. Therefore vulcanization of crude rubber as discussed in this paper means vulcanization with sulfur.

Vulcanization produces definite changes in the rubber. These changes can be defined and frequently measured; yet no good theoretical explanation of vulcanization has been made. It is known that the sulfur combines chemically with the rubber hydrocarbon, but this chemical combination does not explain all that takes place. Furthermore, the rate of chemical combination of sulfur has never been explained fully, since the curve of the rate of addition *versus* time is a straight line function, and the law of mass action seems to have no effect. The proportion of chemically combined sulfur to give soft vulcanized rubber may vary from approximately 0.5 to 5 per cent. A compound with 32 per cent of sulfur chemically combined has all the double bonds known to be in the rubber hydrocarbon saturated, and such a vulcanizate is hard rubber or ebonite. The types of compounding, the use of different accelerators, and different temperatures, all affect the type of final product. The practical generally precedes the theoretical, and this statement is well borne out in the field of rubber.

Optimum Cure:

Properly vulcanized rubber, that is, rubber which has the "best" properties, is said to have the "optimum cure." Such a cure is difficult to define but it ordinarily has the highest tensile strength obtainable with the compound being studied, has good elongation, low permanent set, and good tear resistance. An "undercured" stock, obtained by heating for a shorter time or at a lower temperature, has a low tensile strength, a high elongation, is more soluble than the stock with the optimum cure, shows poor aging, and in general is sleazy. An "overcured" stock, obtained by a longer time of heating or at a higher temperature, may also have a low tensile strength, is "short" (that is, has low stretch), usually tears easily, and generally is rather hard and harsh. As a rule, the stock with the optimum cure gives the best service, including good aging.

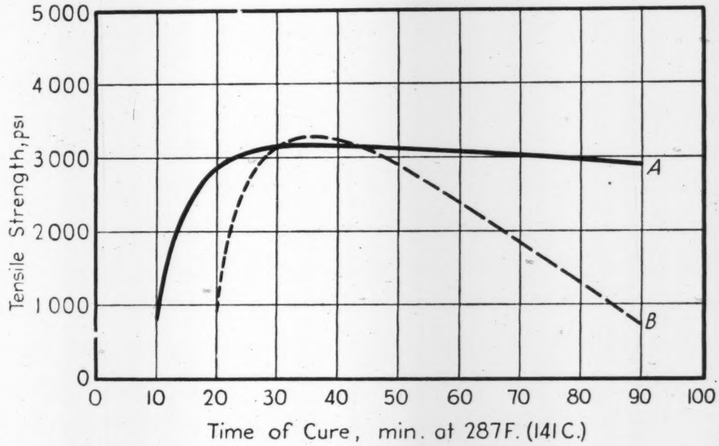


FIG. 2.—Rate of Cure of Natural Rubber—The Desirable "Table-Top" Cure (A) and the Undesirable Type Cure (B).
(*Vanderbilt News*, Vol. 4, No. 6, p. 9 (1934).)

Table-Top Cures:

For many years compounds were generally cured at 141 to 147 C. (40 to 50 psi. steam pressure). However, compounds cured at lower temperatures usually have better physical properties than those cured at the higher temperatures. Certain accelerators can be used at the lower temperatures, about 130 C. (25 psi. steam pressure), and fortunately these accelerators give sustained properties, even though the stock is cured for a rather long period of time. In other words they have a tendency to prevent overcuring, and thus they give so-called table-top cures. The accompanying diagram illustrates this point. The importance of this type of cure is seen in the vulcanization of tires, the outside of which, although actually heated longer at the vulcanizing temperature, is not overcured while the inside layers during the same time are properly cured. Furthermore, for this same reason, it is now possible to cure heavy slabs or large sections of rubber, even though rubber is a poor conductor of heat and the outside is heated for a long period. Since rubber has low thermal conductivity and since vulcanization is an exothermic reaction, when a large or thick object is being vulcanized the temperature must be raised slowly and later lowered slowly over an extended period of time.

Bloom:

Sulfur is soluble in both raw and vulcanized rubber. If the amount of uncombined sulfur in a vulcanizate is greater than its solubility the excess crystallizes throughout the mass and on the surface where it forms a gray finely crystalline powder known as bloom. Bloom does no special harm, but it is unsightly. Therefore, the amount of sulfur and the conditions of cure are adjusted in such a manner that most of the sulfur will be "driven home," and thus avoid the formation of bloom. The use of modern accelerators and careful compounding make this possible.

Protection Against Aging:

Mention has already been made of the aging of rubber compounds. Oxygen and ozone in the air are probably the greatest enemies of rubber, and the use of antioxidants is at present the only known method to combat their action. The conditions under which the rubber is used have much to do with the rate of its deterioration. For example, rubber that is kept slightly stretched, at an elongation of 5 to 20 per cent, will for some unknown reason deteriorate much more rapidly than the same rubber at rest or at higher elongations. It is well known that the portion of a rubber tube that is stretched over a nipple rapidly hardens and cracks. Therefore, rubber used in a bent position, like the edge of a stair tread or an automobile running board, is molded in its final shape in order to avoid the rapid deterioration which would occur under the slight stress in its bent position.

Higher temperatures also increase the rate of deterioration, and special compounding is employed to produce compositions that will withstand them. Much has been learned in recent years about heat-resisting stocks because tires and inner tubes on buses and high-speed trucks now run at much higher temperatures than formerly.

Sun cracking or sun checking is still a major problem which has not been solved completely. Sunlight and ultraviolet light produce a hardening of the surface of rubber goods and the hardening is followed by the cracking of the surface. The action is complex and probably consists of some form of polymerization, auto-oxidation and oxidation with ozone, and also overvulcanization from the absorbed heat. Black compounds are affected more rapidly than light-colored compounds. Sulfur bloom on the surface is somewhat beneficial, but the surface bloom of crystals of hydrocarbon waxes used in the original compounding affords the greatest protection.

Chemical Properties:

As already stated, the rubber hydrocarbon is unsaturated and it contains one double bond for each C_5H_8 group. Unsaturation generally means that the substance is very reactive chemically, but this activity is considerably decreased in substances of very high molecular weight. Both raw and vulcanized rubber have very high molecular weights and are surprisingly inactive toward many strong chemical agents. In some cases the rate of reaction is only slowed up and eventually the total reaction takes place.

Concentrated hydrochloric acid acts very slowly but gradually forms a layer of a hard hydrochloride, while the portion inside retains its characteristic rubber properties. Fifty per cent sulfuric acid shows no apparent action, although acid of higher concentration slowly destroys rubber. Tank cars are lined with rubber for shipment of many corrosive chemicals. Since hard rubber is already chemically saturated with sulfur, it withstands the corrosive action of chemicals to a much greater degree than soft rubber.

Chemical Derivatives:

The chemical unsaturation of rubber is taken advantage of in the preparation of several commercially useful products. Oxidation products in which a comparatively small proportion of oxygen is combined with the rubber hydrocarbon are used as adhesives and in paints. Chlorinated rubber, which contains about 66 per cent of chlorine and is both an addition and a substitution product, finds application in corrosion-resisting paints because of its stability towards strong acids and their fumes. Rubber hydrochloride can be produced in thin transparent sheets which are useful for packaging and for making raincoats.

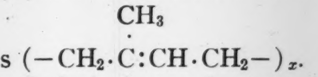
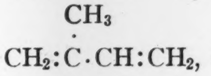
Rubber and Cotton:

There are many cases where the unique stretchability of rubber is not needed, but its resiliency, resistance to abrasion, and impermeability to moisture are needed. In such cases the rubber is supported with cotton fabric. Tires, boots and shoes, tarpaulins, gaskets, diaphragms, hose, and belting are familiar products containing a cotton framework. The cotton imbedded in the rubber, where it is free from the action of air and moisture, has long life and thus can serve its purpose well.

PART II—SYNTHETIC RUBBERS

Synthesis and Structure:

When a naturally-occurring organic substance is synthesized in the laboratory, it is generally not difficult to make a positive identification of the synthetic and the natural. There are, however, substances, especially those of very high molecular weights, which cannot at the present time be identified so definitely. In some cases these substances are probably mixtures, but even when they may consist of a single substance it is exceedingly difficult, if not impossible, to show that the laboratory product is exactly the same as the natural. Such a substance is rubber.

The structure of the rubber hydrocarbon is fairly well, although not completely, understood. Its empirical formula is $(C_5H_8)_x$, and its structure is best represented as  $(-CH_2\cdot\overset{CH_3}{C}:CH\cdot CH_2-)_x$. Since the hydrocarbon isoprene,  $CH_2:\overset{CH_3}{C}\cdot CH:CH_2$, can be obtained from it by heat decomposition, and since rubberlike products can be prepared by heating isoprene in the presence of a catalyst, rubber is considered to be a polymer of isoprene. These synthetic substances are similar to but not identical with the rubber hydrocarbon, and therefore the statement can be made that rubber itself has never been synthesized. And yet in scientific articles, books, and newspapers, and on the radio the term "synthetic rubber" has been used time and again. The explanation of this apparent anomaly lies in the fact that rubber is now also considered as the name of a type of material instead of only a chemical individual. In this sense, rubbers have been synthesized and the term "synthetic rubber" is justified.

Polymers:

Rubber itself and all the synthetic rubbers are polymers, that is, as indicated by the meaning of the term, they are made up of "many parts." Rubber, as stated above, is probably a polymer of isoprene; that is, it consists of the union of a great many molecules of isoprene. Although the laboratory polymerization of isoprene gives a synthetic rubber, it must not be assumed that the rubber tree makes rubber in the same manner, since isoprene has never been found in the rubber tree, and the mechanism of formation of natural rubber is entirely unknown.

Butadiene Polymers:

Isoprene is somewhat difficult to make and is not a commercial chemical, and synthetic rubbers made from it have not been of practical value. However, its chemical parent 1,3-butadiene, $CH_2{:}CH{\cdot}CH{:}CH_2$, is made commercially and is the basic material for several synthetic rubbers. Butadiene is a conjugated diolefin, since it is an unsaturated hydrocarbon containing two double bonds in alternate or conjugated positions. When an unsaturated substance polymerizes, part of the unsaturation always disappears. The polymers of butadiene are large cyclic compounds made up of butadiene residues arranged end to end and having the structure $(-CH_2{\cdot}CH{:}CH{\cdot}CH_2-)_x$, or they are similar long chain compounds with an extra double bond at one end. These polymers may have molecular weights of at least 100,000, and contain upwards of 1800 butadiene residues. Natural rubber varies in molecular weight from 100,000 to 300,000, and therefore probably contains from approximately 1500 to 4500 isoprene residues.

History of Synthetic Rubbers:

Isoprene was first separated by Williams in 1860 as a definite compound from among the products of the destructive distillation of rubber, and fifteen years later its relation to rubber was recognized by Bouchardat who actually converted it to a rubberlike solid. In 1892 Tilden reported that a sample of isoprene which he had prepared from turpentine had spontaneously polymerized to "rubber." Early in the present century Kondakoff found that 2,3-dimethylbutadiene, $CH_2{:}\overset{CH_3}{\dot{C}}\cdot\overset{CH_3}{\dot{C}}{:}CH_2$, a homolog of butadiene and of isoprene, slowly polymerizes to a rubberlike product, and similar observations were made concerning piperylene or 1-methylbutadiene, $CH_3CH{:}CH{\cdot}CH{:}CH_2$, which is an isomer of isoprene, and of butadiene itself. During the first World War about 2500 tons of synthetic rubber were manufactured in Germany from 2,3-dimethylbutadiene. Since this substance is a methyl derivative of isoprene the product was known as methyl rubber.

After the war, work on synthetic

rubber lagged for several years until stimulated by the rise in the price of natural rubber in 1925, caused by the Stevenson restriction scheme and also by the desire of certain countries to be self-sufficient in basic commodities. Research then became very intensive and the several synthetic rubbers on the market today are the results of those activities.

The manufacture of these products depends upon an unlimited source of raw materials, and it is not surprising that petroleum is the chief source with alcohol and calcium carbide as the other sources.

Definition of Synthetic Rubber:

From what has already been given, it is seen that the term synthetic rubber can cover a great many possible materials. At first the term was applied only to substances having chemical structures very similar to natural rubber, but as other rubberlike materials have been produced, even though their structures are very different chemically, it has been extended to cover them also. Then the term rubberlike comes up for explanation. It must be remembered that rubberlike refers not only to natural rubber but to practically all the vulcanized products made from it. These products may be soft and have high stretch and rapid return, or they may be very stiff and have low stretch and slow return depending upon how they are compounded, and yet all are from rubber and their properties have been thought of as rubberlike. Therefore, it can easily be seen why the term synthetic rubber has been used to cover so many types of synthetic materials.

Rubber and all these synthetic materials are plastics, but they are elastic plastics, and that is why they are grouped separately. It is difficult to draw a line of demarcation, since even some of the nonelastic plastics become elastic at temperatures above that of the room. A definition of synthetic rubber therefore should cover their chief property, which is stretchability and rate and completeness of return, with no regard to its chemical composition. How much stretchability and how rapid a return are important questions. A synthetic rubber may be defined as a substance that can be stretched to at least twice its original length and that after having been stretched returns to approximately its original length or position in a reasonable time. The amount of elongation and the statement of the reasonable time of return should be set by committees of national or international organizations before they can be made official.

Nomenclature:

Almost four years ago the author was requested by the editor of *Industrial and Engineering Chemistry* to discuss the nomenclature of the synthetic rubbers and to include a classification of them and new names for them. This was done, and since some of the terms suggested have been used rather widely they are outlined here, although they have not yet been adopted officially. Their derivations are obvious.

All the synthetic rubberlike materials are grouped under the general heading elastomers. Those rubbers which are prepared from butadiene and its derivatives are called elastoprenes. Besides the butadiene rubbers, this term includes natural rubber, since it is theoretically considered to be a polymer of isoprene, and neoprene, which is a polymer of chloroprene or 2-chlorobutadiene. The rubberlike polymers (vistanex) of the simple olefin isobutene are called elastolenes, and the reaction products of ethylene dichloride and alkali polysulfides, known commercially as thiokols, are called elastothiomers. All the others are grouped under the heading elastoplastics, which includes koroseal—a rubberlike, plasticized polyvinyl chloride.

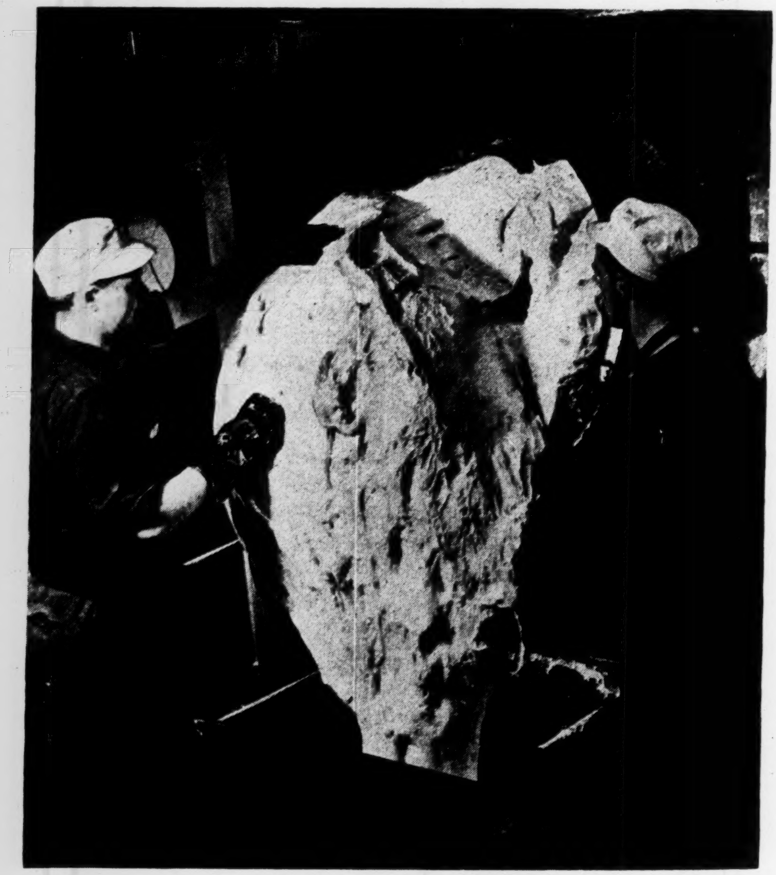


FIG. 3.—Coagulated Neoprene Being Dumped from Polymerization Kettle. (Courtesy of E. I. du Pont de Nemours and Co.)

Composition of Synthetic Rubbers or Elastomers:

As indicated, the synthetic rubbers or elastomers on the market are quite varied in their chemical composition. A few of them are prepared from two separate starting materials which are polymerized in the same operation to form mixed or co-polymers. In these co-polymers the two substances are united chemically in the polymeric chain. Butadiene is generally used as one starting material, and at present the other two materials used commercially are styrene, $C_6H_5 \cdot CH{:}CH_2$, and acrylonitrile, $CH_2{:}CH \cdot CN$. Butadiene and styrene give a synthetic rubber known as buna S, and butadiene and acrylonitrile give products called perbunan or perbunan extra, depending on the proportion of acrylonitrile. Ameripol, hycar, and chemigum are also prepared from butadiene copolymerized with substances whose compositions have not been made public. All of these co-polymers contain high proportions of butadiene. Butyl rubber is a co-polymer in which the butadiene is in the low proportion, and an olefin is in the high proportion. Butyl rubber is not yet commercially available.

The co-polymers of butadiene and the polymers of chloroprene are usually prepared in the form of an emulsion in water in the presence of a catalyst. The final product is therefore an artificial latex. These latices contain particles which on the average are smaller than those in natural rubber latex, and are used in the same general manner for impregnating fabric, etc. The solid elastomer is usually obtained by coagulation with a weak acid just as natural rubber is obtained.

The chloroprene for neoprene rubber is prepared by forming a dimer of acetylene, namely, vinylacetylene, $HC\vdots C\cdot CH:CH_2$, and adding HCl to this. A rearrangement takes place to give chloroprene, $CH_2:CCl\cdot CH:CH_2$. The chloroprene is also polymerized as an emulsion, and the artificial latex obtained is then coagulated to form neoprene. There are several types of neoprene depending upon how they are prepared and made ready for the market.

The reaction product of ethylene dichloride and alkali polysulfides is also obtained as an artificial latex which is coagulated to give thiokol. Thiokol latex has larger particles than those just described. Besides ethylene dichloride other dichlorides are also used, such, for example, as 2,2′-dichloroethyl ether, $ClCH_2CH_2OCH_2CH_2Cl$, which with sodium tetrasulfide gives thiokol B; and glycerol dichlorohydrin, $CH_2ClCH(OH)CH_2Cl$, which gives vulcaplas, an English product.

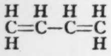
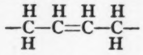
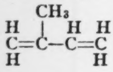
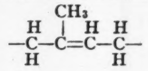
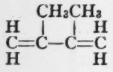
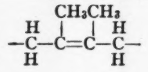
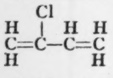
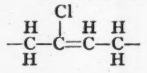
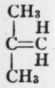
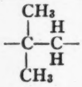
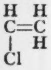
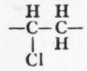
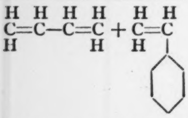
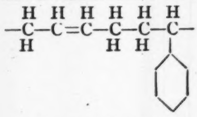
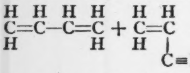
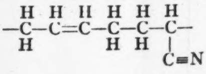
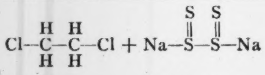
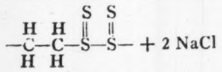
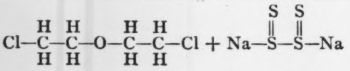
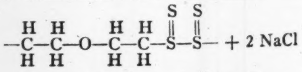
Koroseal is polyvinyl chloride plasticized with a high proportion of tricresyl phosphate or other plasticizer. Vinyl chloride has the structure $CH_2:CHCl$ and can be prepared from ethylene dichloride by the removal of one HCl or from acetylene by the addition of one HCl. Polyvinyl chloride is a white powder with no resemblance to rubber, but when properly plasticized it gives a rubberlike product with many interesting properties.

Table I summarizes part of the above information and gives a more definite idea of the chemical relations of the different starting materials and their polymers. Note the recurrence of the vinyl radical, $CH_2:CH$-, in the compounds.

In Table I, the monomer is the starting material or basic substance, and unit of polymer represents a section of the polymer. The structure of the unit as given has been determined by chemical and physical means but does not show the exact relationship of these units to each other in the chain; in other words they do not always occur in regular order as indicated. In natural rubber there is little doubt that the methyl groups occur regularly, but since isoprene is unsymmetrical, synthetic isoprene rubber has the isoprene groups joined in hit-or-miss fashion with the result that the methyl groups occur irregularly. Furthermore, even in synthetic butadiene rubber which begins, so to speak, with a simple symmetrical molecule, there are units as given, but the polymer is not symmetrical because the structure is complicated probably by the presence of cross-linkings and irregular unions.

Also, the units of co-polymers cannot always be like those given, since the molecular proportions of the starting materials vary considerably. For example, perbunan contains about 7 per cent nitrogen and therefore is made up of about 26 per cent of acrylonitrile. Since the molecular weights of butadiene and acrylonitrile are almost identical,

TABLE I.—CHEMICAL STRUCTURES OF DIFFERENT VARIETIES OF SYNTHETIC RUBBERS.

Monomer		Unit of Polymer
	SIMPLE POLYMERS	
 $\overset{H}{\underset{H}{C}}=\overset{H}{C}-\overset{H}{C}=\overset{H}{\underset{H}{C}}$ 1,3-butadiene	→	 $-\overset{H}{\underset{H}{C}}-\overset{H}{C}=\overset{H}{C}-\overset{H}{\underset{H}{C}}-$ The numbered bunas, SKA, SKB, or ker
 $\overset{H}{\underset{H}{C}}=\overset{CH_3}{C}-\overset{H}{C}=\overset{H}{\underset{H}{C}}$ Isoprene (2-methyl-1,3-butadiene)	→	 $-\overset{H}{\underset{H}{C}}-\overset{CH_3}{C}=\overset{H}{C}-\overset{H}{\underset{H}{C}}-$ Natural rubber
 $\overset{H}{\underset{H}{C}}=\overset{CH_3}{C}-\overset{CH_3}{C}=\overset{H}{\underset{H}{C}}$ 2,3-dimethyl-1,3-butadiene	→	 $-\overset{H}{\underset{H}{C}}-\overset{CH_3}{C}=\overset{CH_3}{C}-\overset{H}{\underset{H}{C}}-$ Methyl rubber
 $\overset{H}{\underset{H}{C}}=\overset{Cl}{C}-\overset{H}{C}=\overset{H}{\underset{H}{C}}$ Chloroprene (2-chloro-1,3-butadiene)	→	 $-\overset{H}{\underset{H}{C}}-\overset{Cl}{C}=\overset{H}{C}-\overset{H}{\underset{H}{C}}-$ Neoprene, Sovprene, or mustone
 $\overset{CH_3}{\underset{CH_3}{C}}=\overset{H}{\underset{H}{C}}$ Isobutene (2-methyl-1-propene)	→	 $-\overset{CH_3}{\underset{CH_3}{C}}-\overset{H}{\underset{H}{C}}-$ Vistanex, Oppanol
 $\overset{H}{\underset{Cl}{C}}=\overset{H}{\underset{H}{C}}$ Vinyl chloride	→	 $-\overset{H}{\underset{Cl}{C}}-\overset{H}{\underset{H}{C}}-$ Polyvinyl chloride Plasticized materials Koroseal, Korogel, or Flamenol
	CO-POLYMERS	
 Butadiene + styrene (vinyl benzene)	→	 Buna S or Buna SS
 $\overset{H}{\underset{H}{C}}=\overset{H}{C}-\overset{H}{C}=\overset{H}{\underset{H}{C}}+\overset{H}{\underset{H}{C}}=\overset{H}{\underset{C\equiv N}{C}}$ Butadiene + acrylonitrile (vinyl cyanide)	→	 $-\overset{H}{\underset{H}{C}}-\overset{H}{C}=\overset{H}{C}-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{C\equiv N}{C}}-$ Perbunan or Perbunan Extra
	ELASTOTHIOMERS	
 $Cl-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-Cl+Na-\overset{S}{\overset{\|}{S}}-\overset{S}{\overset{\|}{S}}-Na$ Ethylene dichloride (1,2-dichloroethane) Sodium tetrasulfide	→	 $-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-\overset{S}{\overset{\|}{S}}-\overset{S}{\overset{\|}{S}}-+2\,NaCl$ Thiokol A
 $Cl-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-O-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-Cl+Na-\overset{S}{\overset{\|}{S}}-\overset{S}{\overset{\|}{S}}-Na$ Dichloroethyl ether Sodium tetrasulfide	→	 $-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-O-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-\overset{S}{\overset{\|}{S}}-\overset{S}{\overset{\|}{S}}-+2\,NaCl$ Thiokol B or Perduren G
	OTHER CO-POLYMERS	

Butyl rubber "is a co-polymer of olefins with small amounts of diolefins."

Chemigum and Hycar OR are co-polymers of butadiene and other substances, the names of which have not been made public.

54 and 53, the molecular as well as the weight ratio is 3 to 1; that is, there are three molecules of butadiene to one of acrylonitrile in the chain. The acrylonitrile must surely be located in irregular fashion along the chain, and besides there may be cross-linkings.

Commercial Production:

The Russians were the first large producers of synthetic rubber. They obtain butadiene from alcohol and petroleum, and polymerize it in the presence of metallic sodium in accordance with the methods discovered independently in 1910 by Harries and by Matthews and Strange. The Russian sodium rubbers are designated by the initials of the Russian words for synthetic rubber followed by A and B depending upon whether the starting materials are petroleum or alcohol, namely, SKA and SKB. Production is said to be around 50,000 long tons a year. The Germans obtain butadiene chiefly from petroleum; some is synthesized from acetaldehyde which in turn comes from alcohol or acetylene. Their early rubbers were produced in the presence of sodium and named "buna" from the first two letters of butadiene and the first two letters of natrium, the German name for sodium. The rubbers formed by polymerization with sodium are given numbers, which refer to viscosities determined under arbitrary conditions: buna 85, buna 115, etc. The Polish product was called ker. In general the sodium rubbers are not as good as natural rubber, whereas the newer types made by copolymerization are often equal and in some respects superior to natural rubber. Germany has a capacity of 40,000 to 60,000 long tons annually and therefore can take care of at least two thirds of her normal rubber requirements.

In the United States annual production of the different synthetic rubbers has been increased from 4000 long tons in 1940 to about 10,000 long tons at the present time. Within a year from this time the annual production is expected to be at the rate of 40,000 long tons a year. How close the industry can come to that amount is problematical. Last year the chief synthetic rubbers were neoprene and thiokol. At the present time neoprene is being produced at the rate of 6000 long tons a year. Thiokol production is around 1500 long tons. The butadiene co-polymers are being produced at the rate of approximately 3000 long tons a year and soon will be produced at the rate of about 5000 long tons.

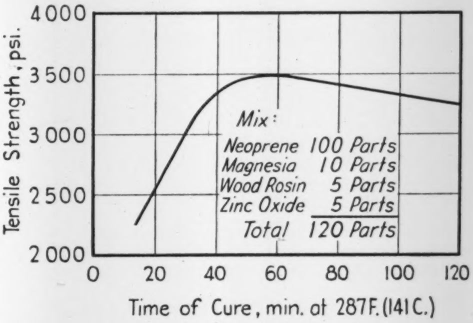


FIG. 4.—Rate of Cure of Neoprene. (*Transactions*, Inst. Rubber Industry, Vol. 14, p. 272 (1939).)

Neoprene manufactured in Russia goes under the name of sovprene, and in Japan under the name of mustone.

Synthetic rubbers are also being manufactured in Italy and England, but the amounts are small.

Vulcanization:

As already stated, the commercially available synthetic rubbers vary in their chemical composition, and therefore it is not surprising that their vulcanizing characteristics vary also. The butadiene co-polymers are nearest to natural rubber in chemical type and they can be vulcanized with sulfur, zinc oxide, and accelerators more or less as natural rubber is vulcanized. Neoprene is also structurally similar to natural rubber, but it has a chlorine in place of a methyl group, and this makes a difference in its vulcanization. Neoprene can be vulcanized with sulfur, but sulfur is not necessary since vulcanization can be effected by heat alone. It is probable that a trace of oxygen is required and the small amount necessary is no doubt always present dissolved in the material. Sulfur in fact, is not always desirable, since it lowers the heat and water resistance of neoprene compounds and tends to promote hardening or aging at elevated temperatures. On the other hand, sulfur improves the resilience and permanent set and this is advantageous since neoprene is inferior to natural rubber in these properties.

Butyl rubber, the co-polymer of a large proportion of an olefin and a small proportion of butadiene, is vulcanized like the other butadiene derivatives with sulfur, zinc oxide, and an accelerator.

The thiokols require a metallic oxide like zinc oxide, and sulfur acts as an accelerator!

Polyisobutene or vistanex is not vulcanizable. It softens with heat and can be molded or mixed with rubber, but it remains a thermoplastic material. Plasticized polyvinyl chloride or koroseal is also a thermoplastic material and cannot be vulcanized. It retains its shape and properties so long as it is not used above 75 C.

When rubber is much overcured a change known as reversion takes place. This change consists of a typical softening of the compound and a general lowering of the desirable physical properties. The synthetic rubbers do not show reversion, and therefore even extra long heating is not disadvantageous but gives the desirable table-top cures mentioned above.

The butadiene rubbers can also be converted into hard rubbers just as natural rubber by the use of a large proportion of sulfur. These hard rubbers have higher softening temperatures and also lower cold flow than those from natural rubber. Neoprene has never been converted into a hard rubber type, and since vistanex, thiokol and koroseal are not vulcanized with sulfur they are not converted into hard rubbers. Mixtures of neoprene, vistanex, or thiokol with natural rubber and high sulfur give hard rubbers that are flexible.

Processing:

Although in general it is true that the synthetic elastomers can be handled on regular rubber mills and other machinery, it cannot be said that they can be handled the same as natural rubber. They are more resistant to air oxidation, and therefore the oxygen of the air does not cause the same breakdown. Also, temperature effects and softening effects with solvents are very different than with natural rubber. Lower temperatures and lower speed ratios of mill rolls, and smaller size of batches are generally required.

Buna S is given a permanent softening by heating it in hot air. This method makes it millable and yet does not appreciably change the properties of the vulcanizates. Perbunan, chemigum, and hycar have a high degree of nerve, and although they can be masticated with a tight mill they are best processed by the addition of special softening agents.

Neoprene can be processed somewhat like rubber with only minor changes, and care is exercised in the order of adding the compounding ingredients to prevent scorching or the stiffening which occurs with premature vulcanization.

Vistanex, thiokol, and koroseal are processed without any great difficulties.

Compounding:

Carbon black and the semireinforcing blacks are the most important compounding ingredients for the synthetic rubbers. Without these blacks the compounder would have considerable difficulty. This statement does not mean that good serviceable compounds cannot be made without the blacks, but the compounds with optimum physical properties are made with them. Carbon black gives compounds with high tensile strength, and good abrasion and tear resistance. The semireinforcing blacks produce compounds having somewhat higher elongations, greater resilience, lower permanent set, and better fatigue and flex resistance but lower tensile strengths than carbon black.

Although the general principles used in compounding natural rubber are applicable to the synthetic rubbers, each synthetic rubber requires special treatments to produce the best results. Besides the milling already discussed, compounding differs somewhat to give proper handling on the calenders and in the extruders, proper tack for building up operations, resistance to heat and abrasion, and other desirable properties.

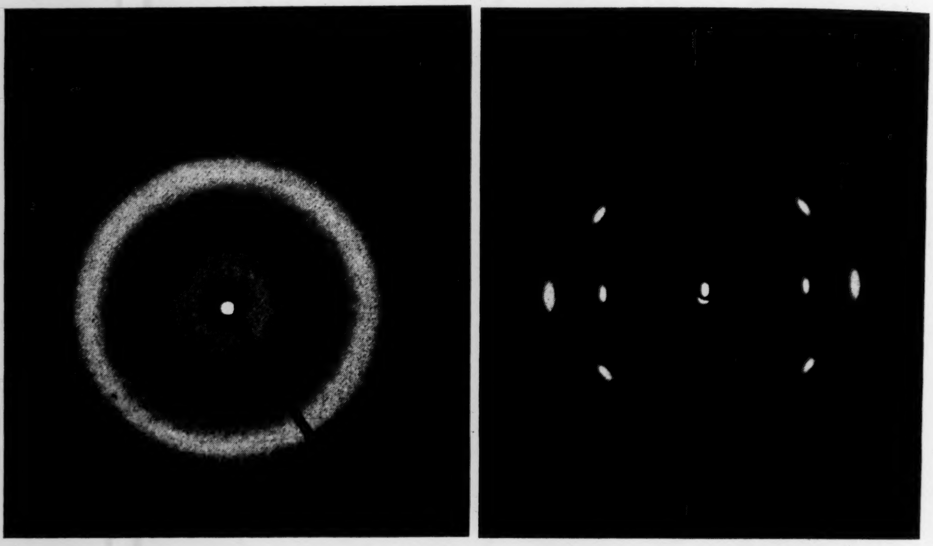
Softeners are used to a greater extent with synthetic rubbers than with natural rubber, since the synthetic rubbers are tougher than natural rubber. In compounds designed for solvent resistance it is advisable, of course, that a softener be selected that will not be extracted by the solvent in service, or, if it is extracted, that the compound be of a type which will still be satisfactory for continued service.

Since synthetic rubbers show much better resistance to aging than natural rubber, it is not always necessary to use antioxidants for this purpose. However, the synthetic rubbers have a tendency to continue to polymerize in storage, and antioxidants have been found to act as retarders of polymerization. The butadiene co-polymers and neoprene generally contain upwards of 2 per cent of an antioxidant, phenyl-*beta*-naphthylamine. In heat-resistant compounds it is necessary to add more of the antioxidant incorporated originally in the stock or to add another antioxidant to further retard the polymerization and prevent hardening.

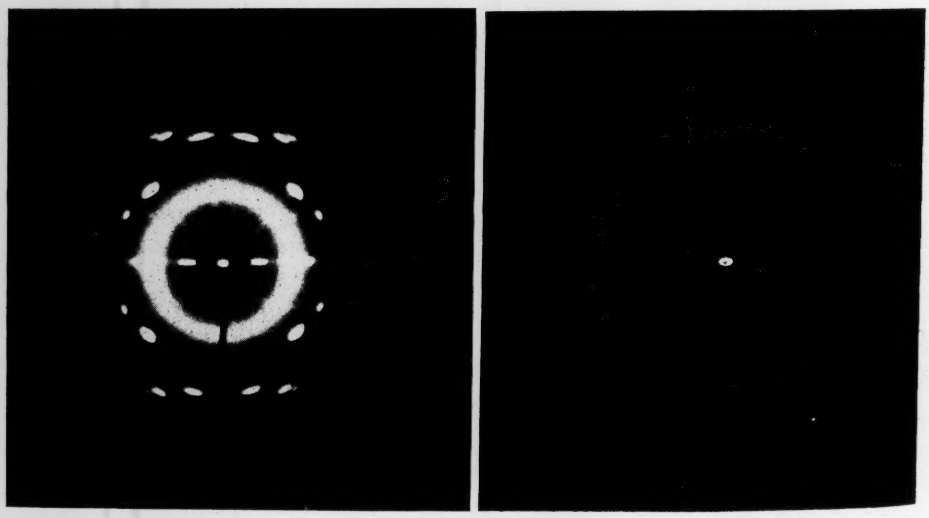
X-ray Structure:

The X-ray diffraction pattern of unstretched natural raw rubber at room temperature consists of a broad halo, which is typical of any liquid. However, when natural raw rubber is stretched above about 75 per cent elongation, there appears a fiber pattern of symmetrical spots which is characteristic of crystalline or definitely oriented structures, and as the elongation is increased, the intensity of the spots increases and the intensity of the halo diminishes. Racked rubber at several thousand per cent elongation gives a pattern of such definite sharpness that it can be concluded that about 80 per cent of this highly stretched rubber is probably crystalline. Vulcanized rubber acts like raw rubber, except that the crystal pattern does not begin to appear until above about 150 per cent elongation. No crystal pattern appears when raw rubber is stretched very slowly, when stretched at temperatures above about 60 C., and when stretched in the vapor of a solvent. Milled raw rubber also shows no crystal pattern even when highly stretched. Unstretched frozen rubber gives a pattern of continuous sharp rings like that of grains of a crystal powder in random orientation.

These are the facts concerning this interesting type of investigation, but the interpretation of them is not so definite. Apparently, when rubber is stretched, individual molecules or portions of molecules become crystalline, at least in part, or oriented as fibers in line with the direction of stretching. The percentage of crystalline material may be calculated for an elongation by measuring the ratio of the intensity of a fiber spot to that of the amorphous halo.



(*a*) Natural rubber, unstretched. (*b*) Natural rubber, stretched.



(*c*) Vistanex, stretched. (*d*) Chemigum, stretched.

FIG. 5.—X-ray Diffraction Patterns.

(*India Rubber World*, Vol. 103, No. 6, p. 37 (1941); Courtesy of Goodyear Tire and Rubber Co.)

On retraction, the ordered or crystalline portion tends to persist, and by plotting the percentage of crystalline material against the percentage elongation, a crystallization hysteresis loop is obtained which is analogous to the usual stress-strain hysteresis loop. A study of the crystallization hysteresis loops has established a direct connection between the degree of crystallinity and creep, extensibility, resilience, and ultimate strength.

What sort of diffraction patterns do the synthetic rubbers give? The buna rubbers give no fiber patterns, only the amorphous halo, whether they are stretched or unstretched. These rubbers may have their constituent groups united in irregular order and also with cross linkages. Synthetic isoprene rubber gives no fiber pattern, probably also because the methyl groups are found in irregular order along the chain. The chlorine atoms in neoprene evidently exercise an orienting influence to bring the chlorine atoms in regular position, since it shows a fiber pattern when stretched. Unsaturation has apparently nothing to do with this phenomenon, since stretched polyisobutene (vistanex) gives a beautiful fiber pattern. Some of the thiokols show a fiber pattern when stretched and some do not.

It had been thought that the results with X-rays would solve the problem of why rubbers stretch. However, the formation of crystallites as shown by X-rays is not a necessary characteristic of rubberlike substances. It is probable that the ability to stretch and retract lies in the alignment of long chain molecules, and that upon release of the stress the relatively ordered arrangement is changed and destroyed possibly by thermal agitation. Also the X-ray diffraction pattern gives no direct information as to the magnitude of the forces involved.

PART III—PROPERTIES AND APPLICATIONS OF SYNTHETIC RUBBERS

In tensile strength, stretchability and speed of comeback, resilience, and lowness of permanent set, natural rubber is still king of the rubbers, but for good aging and for resistance to abrasion, to sunlight, to the action of oils, fats, and solvents, to the coronal discharge, and to the diffusion of gases, the synthetic rubbers have already wrested the crown from natural rubber and in these fields at present stand supreme. Improvements in synthetic rubbers are being made rapidly, and just as the tensile strength and resistance to abrasion have become superior in some synthetic rubber compounds, so other properties may soon be "taken over" and surpass those of natural rubber compounds. Synthetic rubbers are too new to hazard predictions about them. However, it must be remembered that natural rubber is cheap and available under normal conditions, that much more is known about using it than the synthetic rubbers, and that continued work is constantly improving its compounds. A synthetic rubber cannot be substituted directly for natural rubber in a specifically designed compound, but its own characteristics should be considered and used to their full advantage for the desired application.

In a discussion of the applications of synthetic rubbers it would be well to supplement statements of properties with tables of data on many kinds of compounds made up with the several different synthetic rubbers on the market. In the present paper such a procedure is obviously impossible, but the author will endeavor to supply this need partly by giving references in the bibliography below. The whole matter of giving data on compounds is complicated by the great variety of compounds available; therefore, only general statements will be used.

Tensile Strength:

Although tensile strength is not always a necessary criterion of a compound, since each compound should be tested for the service for which it is intended, tensile strength does give an indication of the type of cure and often other desirable properties. A comparison is made in Table II in which the tensile strengths of each material are given, using as examples the unvulcanized materials, and where possible, simple vulcanizates made from them and also vulcanized carbon black stocks, since carbon black gives the maximum tensile strength with synthetic rubbers. The figures are for the maximum tensile strengths in pounds per square inch, and corresponding elongations in per cent, at room temperature. Results vary with different workers but those given are believed to be approximately correct.

TABLE II.—MAXIMUM TENSILE STRENGTHS AND CORRESPONDING ELONGATIONS.

Compound	Unvulcanized		Vulcanized Pure-Gum Compound		Vulcanized Carbon Black Compound	
	Tensile Strength, psi.	Elongation, per cent	Tensile Strength, psi.	Elongation, per cent	Tensile Strength, psi.	Elongation, per cent
Natural rubber	355	1200	4125	710	5000	650
Buna 85	...	...	...	...	2490	600
Buna 115	...	...	...	...	2845	700
Buna S	...	...	...	...	4200	650
Perbunan	...	...	2130	900	5000	600
Chemigum	...	...	...	...	4425	630
Hycar OR	...	...	...	...	3500	450
Methyl rubber W	...	...	425	510	2415	530
Neoprene	425	1100	4265	820	4125	760
Thiokol A	...	...	...	...	855	370
Thiokol D	100	570	500	750	1705	610
Vistanex MM	285	1000	nonvulcanizable		nonvulcanizable	
Vistanex HM	855	1000	nonvulcanizable		nonvulcanizable	
Koroseal, no plasticizer	5690	2	nonvulcanizable		nonvulcanizable	
Koroseal, 30 per cent plasticizer	3840	170	nonvulcanizable		nonvulcanizable	
Koroseal, 60 per cent plasticizer	995	500	nonvulcanizable		nonvulcanizable	

In the above table it is interesting to note that the major increase in tensile strength of natural rubber occurs on vulcanization, and that carbon black only increases the tensile strength somewhat, while it stiffens the rubber considerably. No synthetic rubber compounds have been made with tensile strengths exceeding those of the best carbon black compounds of natural rubber. The butadiene rubbers depend largely on carbon black for strength, whereas neoprene is about as strong without as with carbon black. The thiokols are very weak unvulcanized but gain strength on being vulcanized, especially in the presence of carbon black.

The vistanex with the higher tensile strength also has the higher molecular weight. Koroseal at the medium plasticization shows remarkably high tensile strength, but this decreases with increase in proportion of plasticizer.

Specific Gravity:

Rubber compounds are generally molded, and therefore the specific gravity of the rubber used is of importance, since the amounts of materials are calculated by volume. If a synthetic rubber with a high specific gravity is used in a tire in the same proportion by volume as natural rubber, then the tire will be heavier. The increase in weight may of course be counterbalanced by differences in compounding, but at any rate, changes in weight may require other adjustments in the automobile.

Natural rubber has a low specific gravity, 0.92, and since the butadiene rubbers consist chiefly of hydrocarbons, they also have low specific gravity figures: buna 85, 0.92; buna S, 0.91; perbunan, 0.96; perbunan extra, 0.97; hycar OR, 1.00; and butyl rubber, 0.91. Vistanex also has a low figure, 0.91. Neoprene contains chlorine and has a higher gravity, 1.25; thiokol A is high, 1.60; thiokol D, 1.34; and koroseal with average proportion of plasticizer is 1.30.

FIG. 6.—A Completely Synthetic Rubber Tire. (Courtesy of the B. F. Goodrich Co.)

Modulus:

The term modulus as used in rubber work is the stress at a definite elongation, usually 300 per cent, and therefore is a measure of the stiffness of a stock, since it gives the proportionate resistance to deflection. A relatively high modulus indicates a stiff product and a low modulus a soft, stretchy product. Methods of compounding of course affect the modulus of any rubber stock, but in a general comparison, buna N and especially neoprene show higher modulus figures than natural rubber in corresponding compounds.

Permanent Set:

None of the synthetic rubbers compare with natural rubber in the rate of return after stretching. The set of the synthetic rubber compounds is usually high, although in time the permanent set may compare with that of natural rubber compounds. The elastoprenes, that is, those which are chemically related to butadiene, including neoprene, are more like rubber itself, whereas vistanex, thiokol, and koroseal show a rather high set.

Abrasion Resistance:

Abrasion resistance is an important factor, not only for tires, but also for hose, shoes, and many other common articles. Here again the butadiene polymers compare favorably with natural rubber and in some cases are decidedly better. There are still many problems to be worked out in the processing of the compounds and the methods of handling them in the factory in order to obtain the best results consistently. Since such good results have already been obtained in this country as well as abroad, there seems to be no reason to doubt the complete final solution of the difficulties. Buna S, buna N, chemigum, and hycar OR tread stocks have been shown in tests to be somewhat better than natural rubber, with neoprene close to them. Koroseal is very good for abrasion resistance if no heat is generated, and thiokol is poor.

It should always be emphasized that final judgment must be made on the action of compounds in actual service.

Even with tires, a big difference is shown in the type of driving, since fast driving and buses and trucks produce more heat than conservative driving and smaller vehicles. Also, in cases other than tires, the complete service may depend not only on the resistance to abrasion under normal conditions, but also on how this property is affected by heat, or oils and fats, or the coronal discharge.

Tear Resistance:

Resistance to tear is dependent largely on the type of compound, but in general the synthetic rubber compounds are not as good as those of natural rubber. Carbon black is the best compounding ingredient in all cases to resist tear.

Aging:

Aging has to do with the behavior of rubber compounds under different conditions of air, light, sunlight, and heat, all of which may affect the product at the same time. Laboratory tests are ordinarily made in the oxygen bomb and the air bomb, both at elevated temperatures and pressures, and in heated air in the Geer oven; and the rate of deterioration is estimated by the drop in tensile strength as determined on test strips removed periodically for this purpose.

The synthetic rubber compounds show much better resistance to aging than natural rubber compounds. This statement is true for both laboratory tests and compounds stored or in service under normal conditions, at least so far as it has been possible to observe them for the comparatively short time that some of them have been available. Since natural rubber compounds now show unusual resistance to aging, the better aging of the synthetic rubber compounds means that they are truly remarkable.

As stated above, it is usually not necessary to add antioxidants to compounds made up of synthetic rubbers, but an antioxidant, especially phenyl-*beta*-naphthylamine, is generally added for a different reason, as already mentioned, namely, to prevent hardening by progressive polymerization.

The superiority of the synthetic rubber compounds in ordinary aging is especially noticeable in their resistance to sunlight, which is one of the worst enemies of natural rubber. Sunlight causes checking of natural rubber compounds, as can frequently be noticed in the sidewalls of tires, in the covers of garden hose and electric cables, and in the sponge rubber weather strips around the windows of automobiles. Natural rubber compounds are protected partially by means of waxes which bloom out on the surface, but this method is a makeshift and cannot be used in all cases. Synthetic rubbers are a boon to the compounder who has to design products that are exposed to sunlight.

Temperature Effects:

High temperatures produce a change that is really a part of aging under certain conditions. With natural rubber compounds, as already described, high temperatures produce reversion effects, but with synthetic rubber compounds they show very little effect. In other words, the synthetic rubber compounds have very good resistance to heat. They withstand temperatures of 100 to 150 C. for long periods whereas such temperatures cause decomposition of natural rubber. Long time of heating at these higher temperatures, however, does slowly cause a change, since they become harder and less resilient, probably from a continued polymerization of the constituents. This change is much slower than the decomposition of natural rubber and also is less objectionable. Experiments with some synthetic rubbers have produced unusual results. Under a press, when air was excluded in so far as possible, a sample of perbunan sheet was kept for several days at 200 C. without any noticeable hardening taking place.

Therefore, synthetic rubber compounds are very useful for gaskets, linings of chemical apparatus, hose, and conveyor belts where high temperatures are the rule. In hospital service also they are much in demand because they withstand the necessary frequent sterilizations at high temperatures.

The elasticity of rubbers is related to the temperature. Frozen unvulcanized natural rubber is stiff, opaque, and inelastic, and "melts" and becomes elastic around 5 to 10 C. At the boiling point of water, it is soft, sticky and plastic. This range is much extended by vulcanization, and it is possible to compound natural rubber to keep it elastic for service at −40 C. and above 100 C. The range of synthetic rubbers varies with the type and also with the method of compounding. The synthetic rubbers in general are not as good as natural rubber at low temperatures, although a special neoprene compound has been made for service at −40 C., and butyl rubber is said to be useful at −62 C. There is not much beyond compounding that can be done with natural rubber to change its elastic properties, but synthetic rubbers can be changed by different methods of manufacture of the original product. It is therefore very likely that just as synthetic rubbers have been made to give better service than natural rubber at high temperatures, so they will be made to give better service at low temperatures. The indications are already here, and the definite results will surely follow.

Rubbers for service at low temperatures are very necessary in tires and structural parts in airplanes and automobiles.

Swelling in Solvents, Oils, Fats, and Greases:

The synthetic rubbers are paramount in comparison with natural rubbers in their resistance to swelling in various solvents, oils, fats, and greases. The property of resisting swelling is inherent in the synthetic rubbers themselves, and where the resistance of a particular synthetic rubber is inferior, it usually is not as low as that of natural rubber. As with natural rubber, it can be made better by proper compounding. There are exceptions, of course, since buna S, perbunan, neoprene, and butyl rubber are swelled considerably by benzene and carbon tetrachloride. On the other

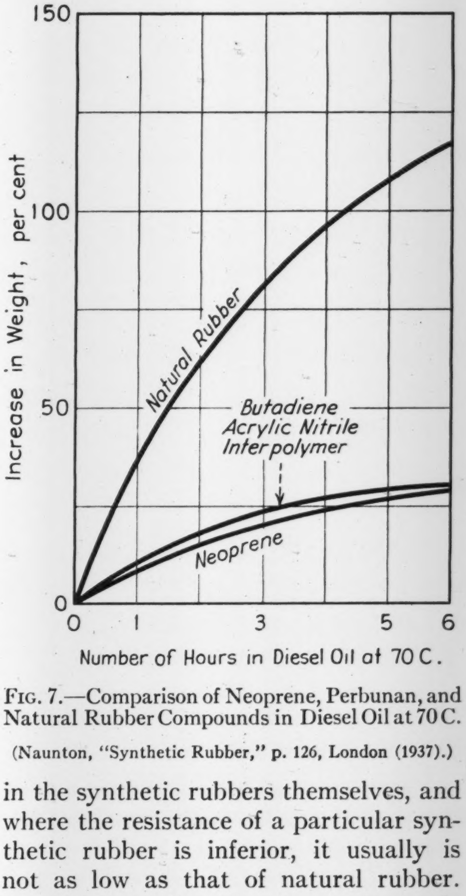


FIG. 7.—Comparison of Neoprene, Perbunan, and Natural Rubber Compounds in Diesel Oil at 70 C.

(Naunton, "Synthetic Rubber," p. 126, London (1937).)

hand natural rubber is not affected by acetone, but perbunan is dissolved by it.

Thiokol A is the outstanding member of the group because its compounds are swelled not at all, or in some cases only slightly, by solvents and oils. Thiokol D is swelled somewhat by aromatic hydrocarbons.

Koroseal also is unaffected by the aliphatic and aromatic hydrocarbons, that is, by gasoline and benzene, but is affected by ketones, esters, nitro and chloro compounds, and amines. These substances remove the plasticizer.

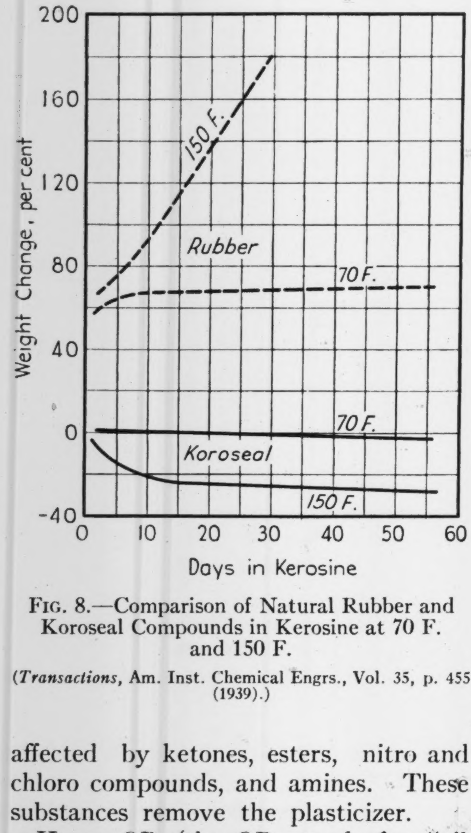


FIG. 8.—Comparison of Natural Rubber and Koroseal Compounds in Kerosine at 70 F. and 150 F.

(*Transactions*, Am. Inst. Chemical Engrs., Vol. 35, p. 455 (1939).)

Hycar OR (the OR stands for "oil resistant") shows excellent resistance to swelling in gasoline, and certain varieties of chemigum are also very good.

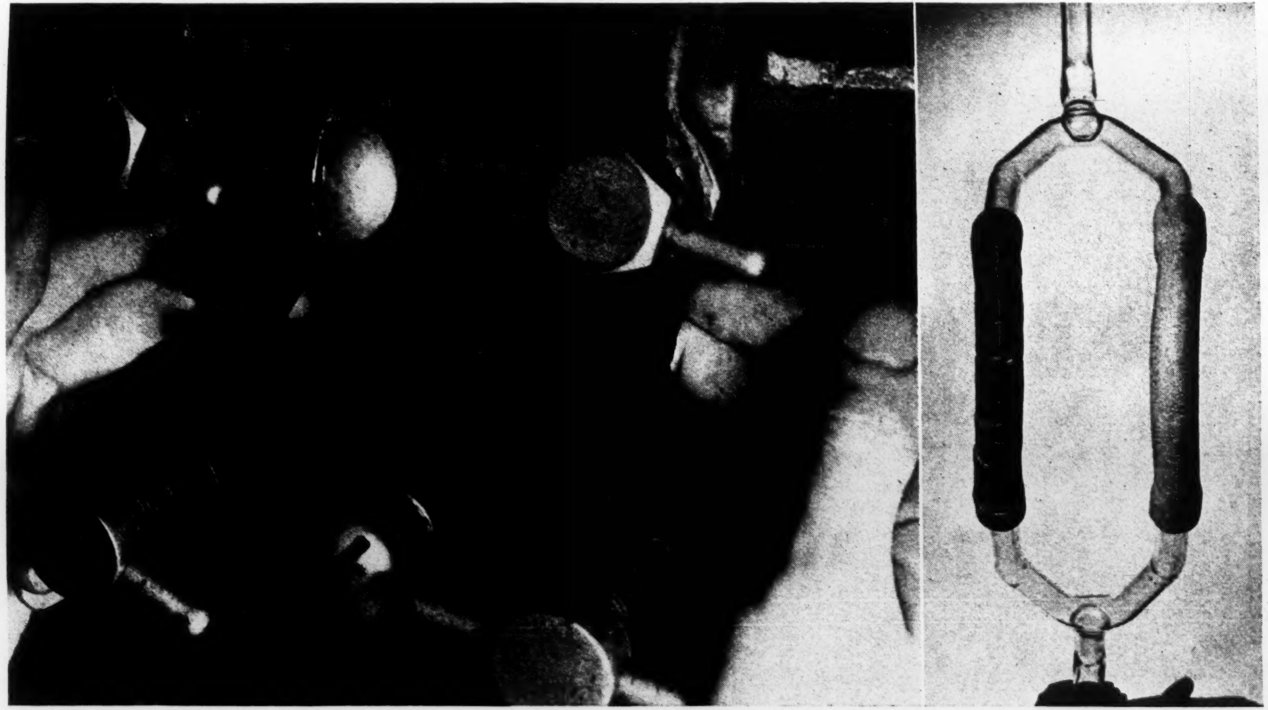
The amount of swelling is not the complete measure of the value of a compound in service, since the real effects are in the changes in elasticity, tensile strength, toughness, and resistance to abrasion. For example, neoprene compounds are swelled in mineral oils, but the actual amount of deterioration is small.

Synthetic rubbers are so useful in their resistance to the action of solvents and oils that, on a quality basis and regardless of price, they are replacing natural rubber in products that come in contact with gasoline, kerosine, lubricating oils, vegetable and fish oils, lard and butter fat, and refrigerating liquids such as freon, methyl chloride, and liquid sulfur dioxide. They also are used to advantage in printer's rolls and blankets, due to their stability and resistance to ink compositions, and in tubes of hose for spraying paints and lacquers.

Effect of Corrosive Chemicals:

Rubber itself, as already stated, is widely used for chemical equipment and for transporting corrosive chemicals, but even though in some cases it can be used for a lining where it is attached to a rigid support, it sometimes loses its elastic properties. Some of the synthetic rubbers are remarkably resistant to the action of very strong acids and oxidizing chemicals, and also retain their elastic properties in long service. Solutions of strongly oxidizing chemicals affect most of the synthetic rubbers except koroseal and butyl rubber, both of which actually withstand the action of concentrated nitric and sulfuric acids. A sample of koroseal was unaffected by a chromium-plating solution during eleven months at 75 C.

Ozone, one of the most destructive chemicals to natural and some synthetic rubbers, does not show much action on neoprene, thiokol, koroseal, and butyl rubber. Such stability is of particular value for insulation purposes where ozone is formed in the surrounding atmosphere by the coronal discharge.



(*a*) After 3,000,000 sharp bends or flexures, a natural rubber compound (left) has been broken, while the butyl rubber (right) is as good as new.

(*b*) Natural rubber tubing (left) and butyl rubber tubing (right) after ozone gas has been passed through them for 8 min.

FIG. 9.—Behavior of Compounds of Natural Rubber and Butyl Rubber Under Flexing and the Action of Ozone.

(Courtesy of Esso Laboratories; photograph by John R. Hall)

Water Absorption:

Although rubber is usually considered as a waterproofing material, it absorbs a small percentage of water when in continuous contact with it. The rubber hydrocarbon is impervious to water, but the protein and other substances in the crude rubber, as well as some compounding ingredients, are water absorptive. Deproteinized rubber has very low water absorption and is the best type for use in electrical insulation. The amount of water absorptive materials in synthetic rubbers can be controlled in their manufacture, and therefore it is not surprising that most of them show very little absorption and correspondingly resist swelling in water.

Electrical Properties:

Soft and hard vulcanized natural rubbers have been used for many years for their good electrical properties, and in recent years these properties have been made much better through the removal of water absorbing substances. The first synthetic hard rubber used commercially was the hard rubber manufactured from methyl rubber in Germany during the first World War. Since its water absorption was very low, it was excellent for use in submarine battery containers and in other electrical equipment. The electrical properties of modern synthetic rubbers depend upon the nature of the original polymers and, of course, upon the compounding. The hydrocarbon polymers, namely, the numbered bunas, buna S, vistanex, and butyl rubber, are superior to natural crude rubber, whereas those containing polar groups—neoprene and koroseal, which contain chlorine, and perbunan, which contains nitrile groups—are poorer. This is a very general statement and is only relative, since neoprene and koroseal are important materials for flameproof, oil-resistant electrical insulation.

It is difficult to discuss electrical properties without using figures, and only a few general results will be given here. Each adaptation requires specific measurements, and some of these will be found in the papers listed in the bibliography. Compounding changes all the figures cited.

The dielectric constant of natural rubber is around 2.35 at 1000 cycles per sec., and of compounded natural rubber 3.0 to 5.0. For the hydrocarbon polymers it may be lower than for natural rubber, 2.11 to 2.9; it is higher for thiokol, 4.6; 5.72 for koroseal with 30 per cent of plasticizer; 7.5 for neoprene; and 15 for perbunan.

The power factor of crude rubber is around 0.003 and of vulcanized compounded rubber, 0.004. The figures for the hydrocarbon polymers are similar to those for natural rubber; they are higher for thiokol, 0.010, and neoprene, 0.193, and lower for vistanex, 0.0015.

The direct-current conductivity of crude rubber varies greatly, depending upon the nonhydrocarbon constituents. In terms of reciprocal ohms, for deproteinized rubber it is very low, 1.6 to 3.3 mho per cm. × 10^{-17}; pale crepe, 25; and Fine Pará, 29. The synthetic rubbers, excepting vistanex, exhibit a higher conductivity than natural rubber. The corresponding figure on a comparison basis is not less than 100 for the numbered bunas and buna S, 100,000 for neoprene, and 100,000,000,000 for perbunan. For vistanex it is less than 2. The absorption of water generally increases the conductivity of natural and synthetic rubbers.

The dielectric strength of the synthetic rubbers varies from below to above that of natural rubber. Vistanex and koroseal are greater in this property, and thiokol and neoprene lower, although they are within a practical range.

Since buna S and vistanex show superior electrical properties to natural rubber, they are finding application as insulation for wires and cables. Neoprene, thiokol, and koroseal are also used for insulation, especially at low voltages. Furthermore, since they are resistant to sunlight, ozone, and oils, they are very valuable as jackets over insulation of natural rubber or other materials. They are replacing lead sheaths and are preferred on nonleaded, rubber-insulated cables, which are usually installed on poles between overhead and underground circuits.

Elasticity, Hysteresis-Loss, Compression-Set, etc.:

These are subjects which should require an entire paper for proper discussion, but only a few general statements can be made.

All the synthetic rubbers are more or less elastic; that is why anyone will readily call a sample of them, rubber. The elastic properties vary with the temperature. Polystyrene, a plastic substance, is nonelastic at room temperature but becomes elastic above about 60 C. Some synthetic rubbers are nonelastic at temperatures below those of the room, and some remain elastic at higher temperatures at which rubber begins to soften.

Natural rubber compounds are superior to synthetic rubbers in elasticity and they show the lowest energy loss due to hysteresis, and the lowest rate of heat build-up under rapid deformation. Next to natural rubber comes neoprene GN, while the buna types are lowest in elasticity and highest in hysteresis loss. Compounds of natural rubber generally show superior energy and lower permanent set than the corresponding compounds of synthetic rubber. These properties are all very important considerations in the mechanical applications of natural and synthetic rubbers.

Synthetic rubbers generally show a greater absorption of energy during repeated stresses, and this quality is desirable where rapid damping of vibrations is important, although in a tire it may lead to undesirable overheating. Like liquids, rubber has extremely small cubic compressibility, but if the sample is not entirely confined, a force acting upon it will of course deform it and upon release it will regain part of its original shape. Compression-set tests show that synthetic rubbers give results which fall both above and below those of natural rubber.

Dynamic tests prove that natural rubber shows greater resilience than any of the synthetic rubbers. At temperatures above 100 C., however, balls of butyl rubber give approximately the same rebound.

Much work is being done on the use of natural and synthetic rubbers in engine mountings, rubber springs, parts for absorbing vibration, etc., where permanent set, resilience, creep in shear, as well as aging and oil resistance, must be considered and measured. Modern materials and recent developments have opened up many fields in which these new rubbers are finding useful applications.

Diffusion of Gases:

The diffusion of gases through synthetic rubber compounds is at a much lower rate than through natural rubber. This has been proved with hydrogen, helium, nitrogen, air, and sulfur dioxide. The permeability to hydrogen diminishes in the following order: natural rubber, perbunan, neoprene G, vistanex, thiokol DX, koroseal. This low permeability to gases, together with the stability of the compounds in direct sunlight, have made synthetic rubbers attractive for use in a field where natural rubber always had held sway. Small balloons for meteorological observations and the envelopes of large passenger-carrying dirigibles are now made of these new products. On the other hand, in the build-up of synthetic rubber compounds in factory operation, this low permeability makes it important that all air be eliminated between the plies.

Flexing:

The nature and conditions of the flexing have much to do with how long a compound will last. In a regulation laboratory test koroseal was serviceable after 3,000,000 flexures, while natural rubber failed at 300,000. In another experiment a natural rubber compound failed at 850,000 flexures and butyl rubber not until 5,000,000. In tread compounds natural rubber is still at the top, although some of the synthetic rubbers compare favorably. Flex-cracking is important in tires, power belting, shoes, hose, insulated wire, and many other articles.

Flame Resistance:

Natural rubber alone and in its compounds burns when a flame is applied and continues to burn when the flame is removed. The butadiene polymers and thiokol are similar to natural rubber, but neoprene and koroseal are flame-resistant and usually do not continue to burn when the flame is removed.

Color:

Koroseal can be compounded for any desired color. Natural rubber can be used similarly, but the colors are somewhat less bright and tend to darken upon aging. Neoprene is lower in color range, and thiokol is still lower. The color of these rubbers is usually due to the presence of antioxidants which darken in the air and light. Color in many mechanical goods plays no part in their use, and this is fortunate, since the best properties of synthetic rubbers are brought out by compounding them with carbon black and the semireinforcing blacks.

Odor:

The odor of natural rubber goods is familiar to everyone. By removing the protein material and by proper choice of compounding ingredients this odor can be practically eliminated. Koroseal is free from odor, but most of the other synthetic rubbers have definite persistent odors.

Adhesion:

By the use of proper milling and compounding or of proper cements natural rubber readily adheres to itself and can be made to adhere to practically anything else. Similarly, the synthetic rubbers in general can be made tacky enough to adhere to plies of their own selves and also to natural and other synthetic rubbers. The difficulties have been great, and there are serious problems yet to be solved. However, the synthetic rubbers can be had with backings of metals, hard rubber, plastics, and fabrics, for use in a great variety of applications.

Reclaiming:

The rubber industry has applied a sound principle of economics by saving and working over for use articles that have been worn out. Approximately 200,000 tons of reclaimed rubber are prepared annually, and this amount is being increased at the present time. Reclaimed rubber is not the same as raw rubber, although it can be vulcanized. It is replasticized vulcanized rubber, and is a very important compounding ingredient.

The vulcanized synthetic rubbers are different chemically from vulcanized natural rubber, but the replasticization of some of them has already been worked out and replasticization of the others will no doubt follow in due season. The difficulties do not appear to be insurmountable.

Uses of Synthetic Rubbers Where Natural Rubber Could Not Be Used:

The early workers on synthetic rubber tried to duplicate the natural rubber hydrocarbon. They did not dream of making rubbers that would be applicable where natural rubber just does not have a chance, and yet that is what has happened. These new applications require resistance to gasoline, oil and other solvents, and to abrasion, flame, heat, and sunlight. A few examples will serve to illustrate these new uses.

Perhaps the most striking examples and the most interesting at the moment are the fabrication of fuel cells for military aircraft. One type consists of a "sandwich" of synthetic elastomer, unvulcanized natural rubber, and reinforcing materials. In case a bullet bores a hole through the sandwich, the swelling of the inner layer of natural rubber by the gasoline closes the hole while the gasoline-resistant elastomer keeps its shape, and thus the entire cell is maintained intact. The other type is the Mareng fuel cell, which consists of a bag of fabric impregnated with a gasoline-resistant synthetic elastomer and enclosed by a metal container; it is proof against vibration and corrosion.

Another vital use is for the diaphragms of the new aircraft carburetors which utilize a synthetic elastomer assembly instead of a float mechanism, and a variable venturi in place of the conventional butterfly valve and fixed venturi. Since the diaphragm chamber is always entirely filled with fuel when the engine is running, there is never any trouble from splashing, even when a plane is flown upside down.

Much of the improvement in deep oil well drilling is due to the design of equipment that is dependent upon the use of these new oil- and heat-resistant elastomers. Great strides have been made in the printing industry through the use of rolls and draw-sheets made of the new elastomers, whereby it is possible to use the new quick-drying ink vehicles which would destroy natural rubber. Heat-resistant elastomers are used in conveyor belts for carrying hot slag, and similar oil-resistant types are used in conveyor belts for oil-treated coal.

The success of airplane de-icers depends upon the neoprene veneer which protects the underlying rubber from deterioration in sunlight.

The synthetic elastomers find application in mine and shipboard power cables where it is essential that materials be used that do not propagate fire.

Other applications are for hydraulic press gaskets for use in hot service, for gaskets where oil replaces water in liquid pressure systems, for gasoline hose nozzles, core drill bearings, rod packings, protective clothing for the chemical and petroleum industries, flexible ebonite for use in structural parts of chemical equipment, sealing members on reciprocating pistons, transformer gaskets, and tubing and diaphragms for handling chlorine gas, ozone, and sulfur chloride.

SUMMARY AND CONCLUSIONS

Rubber is a very versatile material and, now that its "life" has been lengthened, its interesting and varied properties are making possible the enormous extension of its usefulness. Even though the greatest use of rubber is in tires and tubes for automobiles, more and more rubber is being used in the structural parts of the automobile, and, in fact, today the unique properties of rubber are literally forcing its use in structures of all kinds of products.

Natural rubber has certain drawbacks and is meeting competition on a quality basis from the new synthetic rubbers. The chemistry and physics and the methods of compounding these interesting materials are briefly discussed and compared with those of natural rubber.

Vulcanized synthetic rubber compounds resemble natural rubber compounds in the following properties:

1. Appearance,
2. Elasticity and extensibility,
3. Resilience,
4. Tensile strength,
5. Abrasion resistance, and
6. Electrical properties

Vulcanized synthetic rubber compounds are superior to natural rubber compounds in:

1. Aging in storage and sunlight,
2. Resistance to the action of air and ozone,
3. Resistance to deterioration by heat,
4. Resistance to the action of solvents, oils, fats, and greases,
5. Resistance to many corrosive chemicals,
6. Water absorption, and
7. Permeability to gases.

Natural rubber compounds are still superior to all synthetic rubber compounds in:

1. Elasticity and rebound,
2. Low energy loss and low heat generation through hysteresis,
3. Extensibility, and
4. Resistance to stiffening at low temperatures.

In the earlier days of the rubber industry, the compounder was confronted with many different varieties of natural rubber obtained from different parts of the world. At present he has only about three varieties of natural rubber—pale crepe, smoked sheet, and amber—but he has several different types of synthetic rubbers and different varieties of each type, all of which show much greater differences in properties than the varieties of natural rubber, and require different methods of handling and compounding. The compounder, however, has an easier task, from one point of view. With natural rubber he has worked hard to put it into places where its properties were against its use, but now he can choose the right synthetic rubber for these difficult jobs and have that comfortable feeling of assurance that it will work.

At the present time in this country, synthetic rubbers are making headway on the basis of their outstanding properties, regardless of the higher costs involved. If the supplies of natural rubber should be cut off, synthetic rubbers will of course be tried out in many places where ordinarily they would not be considered. Then, when the war is over, even though the prices of synthetic rubbers may still be higher than those for natural rubber, their use in many cases will no doubt be continued—remember the fable of the camel who at first got only his head under the bottom of the tent of his keeper.

Acknowledgment:

Several months after the author was asked to give this paper, there appeared two papers covering some of the material originally planned for this one, and he is glad to acknowledge his indebtedness to the authors of them: J. W. Schade, and L. B. Sebrell and R. P. Dinsmore. Their papers are included in the bibliography. An excellent summary of

published work on synthetic rubbers was issued a short time before by Lawrence A. Wood of the National Bureau of Standards and the author is glad to acknowledge his indebtedness to this work also. He is especially grateful to O. M. Hayden for his interest and thoughtful suggestions.

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COMPRESSION AND TENSION TESTS OF STRUCTURAL ALLOYS

By Bruce Johnston[1] and Francis Opila[2]

Synopsis

Tension and compression tests were made on 33 samples of carbon, silicon, and low-alloy structural steels. Two samples of structural aluminum alloy 27S-T were also included in the program. All samples except one were tested both with and across the direction of rolling. The tests included the determination of modulus of elasticity and Poisson's ratio, the proportional limit, the upper and lower yield points, the stresses at maximum and breaking loads, the usual ductility criteria, and the "true" ductility indices proposed by MacGregor.[3]

Special tests were made to illustrate the effect of different testing techniques used in compression and tension. Tests were also included to illustrate the "Bauschinger" effect resulting from successive cycles of tension and compression into the yield point range.

A comparison was made between tensile and compressive stress-strain characteristics together with special study of directional properties with and across the direction of rolling. The general shape of the stress-strain diagrams and the values of upper and lower yield point were found to average nearly the same in tension as in compression. Silicon structural steel was found to have the greatest directional variation and the least ductility of the steels tested. The elastic constants were found to be not affected by the direction of rolling. The most definite directional properties were found to be associated with the behavior of the material beyond the maximum load.

The need for further information on the subject of the compressive stress-strain characteristics of structural metals, particularly of low-alloy and silicon steels, was suggested to the authors by members of the American Society of Civil Engineers Committee on "Design of Structural Members." Such knowledge is fundamental to the design of plate elements or structural sections subjected primarily to compressive stresses. Accordingly, several companies were invited to submit a variety of materials, which finally included eleven different heats of carbon structural steel, seven of silicon structural steel, fifteen of various low-alloy high-strength steels, and two samples of structural aluminum alloy 27S-T. At least four different tests were made from each sample, these being tension and compression tests both with and across the direction of rolling. As much information as practicable was obtained from each test and in the case of the tension tests use was made of suggestions[3] presented in the

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[3] C. W. MacGregor, "The Tension Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 508 (1940).

1940 A.S.T.M. "Symposium on Significance of the Tension Test of Metals in Relation to Design." MacGregor's and other papers of the symposium include rather complete bibliographies on the tensile and related properties of structural materials. A theoretical discussion of the various phenomena associated with the stress-strain characteristics of structural metal is outside the scope of this paper. Many of these phenomena, such as the upper and lower yield point in structural steel, are not yet fully understood and controversial hypotheses exist. There are available numerous reviews and compilations of existing knowledge on these subjects among which the writings of Nadai,[4] MacGregor,[3, 5] and Gensamer[6] provide sources readily available in this country.

Definitions of the terms used in this paper follow:

Unit Stress.—Unit stress in the tension or compression test is denoted by the Greek letter σ and is the average stress intensity in the tension or compression specimen. Nominal stress is that obtained by using the original area A_o, and is denoted by $\sigma_N = \frac{P}{A_o}$. True stress is the average tensile or compressive stress over the minimum or maximum actual area, respectively, at any particular load and is denoted by $\sigma_T = \frac{P}{A}$.

Strain.—Longitudinal strain is herein denoted by the Greek letter ϵ and is defined as the average deformation per unit original length over a 1-in. gage length on the 0.505-in. or 0.500-in. specimens used in the tension and compression tests. The notion of "true" strain is used in connection with "true" ductility indices in the manner presented by MacGregor.[3]

"True" strain was first defined as such by Ludwik[7] and is denoted by

$$\epsilon = \int_{L_o}^{L} \frac{dL}{L} = \log \frac{L}{L_o} \quad \ldots\ldots \quad (1)$$

where L_o is the original gage length and L is the actual length at any particular load. When local necking commences in a tension test, the strain becomes nonuniform within the usual gage length. On the very nearly correct assumption that the volume change is zero in the plastic range, the maximum true strain in the most reduced section may be determined as

$$\epsilon = \log \frac{A_o}{A} \quad \ldots\ldots\ldots \quad (2)$$

Equation 2 follows directly from Eq. 1 and the assumption of zero volume change. A more complete discussion of true strain is presented in the 1940 A.S.T.M. *Proceedings*.[3]

Elastic Moduli.—Elastic moduli are experimental relationships between stress and strain. At stresses below the proportional limit these relationships are constants and as such are evidences of Hooke's law. Young's modulus of elasticity (E) as determined by these tests, is the ratio below the proportional limit of average stress divided by longitudinal strain in the tension or compression test. Poisson's ratio (ν), is the ratio between lateral and longitudinal unit strain below the proportional limit. These two elastic constants are sufficient to relate the most general states of stress and strain for the case of isotropic materials.

Proportional Limit.—The proportional limit (σ_P) as determined in this investigation is the maximum stress below which the stress-strain graph deviates from the initial straight-line relationship by a strain of less than about 1/100,000.

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[4] A. Nadai, "Plasticity," McGraw-Hill Book Co., Inc., New York, N. Y. (1931).

[5] C. W. MacGregor, "The Yield Point of Mild Steels," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 53, pp. 187–200 (1931).

[6] M. Gensamer, "The Yield Point in Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 128, pp. 104–114 (1938).

[7] P. Ludwik, "Elemente der Technologischen Mekanik," Julius Springer, Berlin (1909).

Upper Yield Point.—The phenomenon of the upper or "drop-of-the-beam" yield point is a characteristic of structural steels. Upper yield point is the stress (σ_{UY}) at which a pronounced increase in strain is accompanied by a falling off of the load.

Lower Yield Point.—The lower yield point (σ_{LY}) is the lowest stress at which a slow, continued increase in average strain is maintained after passing the upper yield point. The plastic strains in structural steels while the lower yield point stress is just maintained may range between ten and twenty times the elastic strain corresponding to the yield point load.

Yield Strength.—Some steels and most non-ferrous metals exhibit no well-defined upper or lower yield points and for such materials the term "yield point" is meaningless. The term "yield strength" is sometimes applied to such materials and is the stress corresponding to an arbitrary offset from the straight part of the stress-strain curve. For materials such as structural aluminum alloy the Society specifies the offset as 0.2 per cent.[8]

Measures of Ductility.—At loads less than maximum the tension test specimen undergoes a uniform reduction of area and plastic elongation all along the specimen. After the maximum load is passed the further reduction of area and plastic elongation are localized in one particular region and "necking down" takes place. The usual percentage reduction in area and percentage elongation in 2 in. represent a combination of the "uniform" and "necking" phases of plastic yielding. The "true" indices of ductility proposed by MacGregor[3] differentiate the "uniform" and "necking" phases and at the same time correlate reduction in area with elongation, since the two become numerically equal. The "true" ductility indices are obtained simply by determining the least area of the tensile specimen initially (A_o), at the maximum load (A_u), and after fracture (A_b). The true indices of ductility are then defined as: Uniform ductility index (ϵ_u) = true strain between zero and maximum load = $\log \frac{A_o}{A_u}$. Necking ductility index (ϵ_n) = true strain at most reduced section between maximum and breaking load = $\log \frac{A_u}{A_b}$. Over-all ductility index (ϵ_b) =

TABLE I.—CHEMICAL ANALYSES OF STEELS.

Sample	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent
No. 1	0.17	0.57	0.012	0.030	0.07
No. 2	0.15	0.44	0.012	0.024	0.01
No. 3	0.16	0.44	0.012	0.024	0.01
No. 4	0.19	0.48	0.012	0.015	0.06
No. 5	0.18	0.46	0.013	0.026	0.06
No. 6	0.22	0.33	0.015	0.037	0.01
No. 7	0.18	0.33	0.012	0.033	0.01
No. 8	0.17	0.63	0.012	0.030	0.07
No. 9	0.24	0.45	0.016	0.033	0.01
No. 10	0.25	0.45	0.012	0.040	0.03
No. 11	0.21	0.50	0.018	0.028	0.15
No. 12	0.28	1.07	0.020	0.020	0.24
No. 13	0.28	1.06	0.020	0.024	0.25
No. 14	0.35	0.96	0.012	0.028	0.27
No. 15	0.30	0.94	0.012	0.025	0.20
No. 16	0.32	0.82	0.016	0.030	0.25
No. 17	0.34	0.88	0.019	0.035	0.35
No. 18	0.28	1.00	0.023	0.025	0.25

TYPICAL ANALYSES OF LOW ALLOYS

Sample[a]	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Nickel, per cent	Chromium, per cent	Copper, per cent
No. I	0.14	0.34	0.104	0.033	0.36	0.56	0.80	0.39
No. II	0.10	0.64	0.085	0.024	0.28	0.43	0.53	0.61
No. III	0.17	0.50	0.030	0.023	0.05	1.83	0.21	0.63
No. IV	0.26	1.33	0.018	0.027	0.01	0.03	....	0.21
No. V	0.19	0.73	0.018	0.026	0.16	1.78	0.01	1.11
No. VI	0.20	0.82	0.014	0.036	0.29	0.53	0.57	0.50
No. VII	0.09	0.95	0.110	0.022	0.02	0.14	0.04	0.29
No. VIII	0.07	0.31	0.012	0.028	0.01	1.84	....	1.14

[a] These sample numbers are purposely not correlated with the test results in Table II, as requested by several manufacturers.

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[8] 1939 Book of A.S.T.M. Standards, and 1940 and 1941 Supplements to the Book of Standards, Part I.

total true strain at most reduced section between zero and breaking load = $\log \frac{A_o}{A_b} = \epsilon_u + \epsilon_n$.

MATERIALS

All of the samples, with two exceptions, were taken from plate stock. Table I gives the chemical analyses of all of the carbon structural and silicon structural steels in the program. Typical analyses of the eight different types of low-alloy steels are also furnished in Table I, but these are not correlated with the listings of samples in Table II on test results.

The direction of rolling of the plate material as designated by the manufacturer was checked in the laboratory by macroscopic examination. This was accomplished by lightly polishing and hot etching with hydrochloric acid on the rolled surface. In most cases the direction of final rolling was readily checked by this procedure but in some cases the surface had an isotropic appearance and the manufacturer's designation was accepted.

A minimum of 1 in. of material was cut away from all burned or sheared edges before machining the test specimens. The rough cuts on the structural aluminum samples and on most of the steel samples were made by band saw.

APPARATUS, TEST METHODS, AND SPECIAL TESTS

In making the tension tests the threaded-end specimen specified in the A.S.T.M. Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T)[9] was adopted. The specimen was made with a gage length of 2 in. and a diameter of 0.505 in. The fillets on the specimen were made with a radius of approximately ½ in., and the ¾-in. diameter ends had ten threads to the inch. All tests were made on a Baldwin-Southwark 60,000-lb. hydraulic testing machine.

The usual type of gripping device for the threaded test specimen consists of a shaft with a threaded recess at one end and a ball-and-socket joint at the other end which fits a socket in the head of the testing machine. In many cases the socket is formed by a split wedge. When the two parts of the wedge are seated unevenly, a considerable eccentricity is introduced into the specimen.

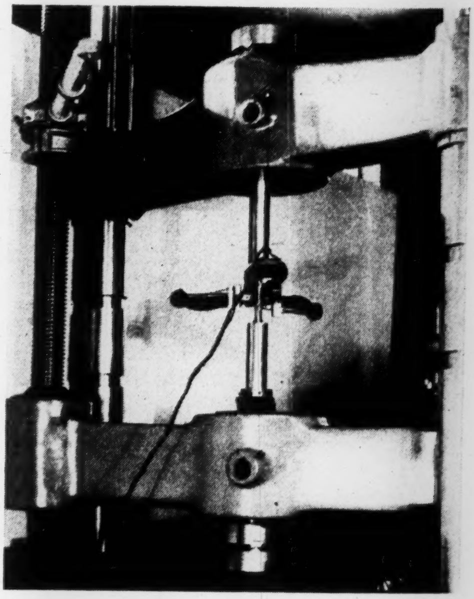

FIG. 1.—Apparatus Used in Tension Test.

In order to avoid the possibility of friction in the ball-and-socket device, and to insure quick adjustment for load axiality, a knife-edge loading device was designed as suggested by work done in England by J. G. Docherty and F. W. Thorne.[10] Two sets of knife-edges were inserted into steel plates and mounted

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[9] 1940 Supplement to Book of A.S.T.M. Standards Part I, p. 453.

[10] J. G. Docherty and F. W. Thorne, "The Phenomenon of Tensile Yield in Mild Steel and Iron," *Engineering*, Vol. 132, September 4, 1931, p. 295.

TABLE II.—GENERAL TEST RESULTS.

Steel	Test Number	Source	Tensile Stress, 1000 psi.						Compressive Stress, 1000 psi.					Ratio, σ_{UY} to σ_{LY}		Ratio, σ_{UY} to σ_{NM}		Ratio Compression/Tension		Elongation in 2 in., per cent	Reduction of Area, per cent	Ductility Indices			Brinell No.	Elastic Constants			
			σ_P	σ_{UY}	σ_{LY}	σ_{NM}	σ_{TM}	σ_{TB}	σ_P	σ_{UY}	σ_{LY}	σ_{NM}	σ_{TM}	Tension	Compression	Tension	Compression	σ_{UY}	σ_{LY}			Uniform	Necking	Total		E_t, 1,000,000 psi.	E_c, 1,000,000 psi.	ν_t	ν_c
Carbon Structural	1A	¾-in. plate	36.6	37.6	34.7	61.7	80.8	103.6	28.6	36.2	34.5	66.1	61.6	1.08	1.05	0.61	0.55	0.96	0.99	38.0	58.7	0.268	0.616	0.884	116	29.9	29.6	.....	.....
	1B		28.8	36.8	32.8	61.1	78.8	120.4	32.4	35.5	32.7	66.1	61.2	1.12	1.08	0.60	0.54	0.97	1.00	40.0	63.8	0.254	0.761	1.015		29.6	29.6	.....	.....
	2A		29.9	32.4	30.7	56.7	70.7	114.3	28.5	32.2	29.4	61.0	56.8	1.05	1.09	0.57	0.53	0.99	0.96	41.5	64.3	0.221	0.806	1.027	101	29.6	28.9	.....	.....
	2B		26.2	32.7	28.2	55.0	62.9	104.6	28.5	31.7	28.9	61.0	56.6	1.16	1.10	0.60	0.52	0.97	1.02	28.0	60.7	0.133	0.804	0.937		29.5	29.4	.....	.....
	3A		26.8	33.0	29.5	56.0	70.1	111.8	30.4	32.2	29.4	55.8	52.6	1.12	1.10	0.59	0.58	0.98	0.99	45.0	64.2	0.223	0.806	1.029	98	29.5	29.2	.....	.....
	3B		25.6	33.0	30.4	55.7	69.6	102.6	31.2	32.1	29.6	61.0	56.6	1.09	1.09	0.59	0.53	0.97	0.97	43.5	59.1	0.222	0.673	0.895		29.5	29.5	.....	.....
	4A		26.2	41.9	39.7	64.7	78.3	121.0	25.4	41.0	39.1	69.8	66.7	1.05	1.05	0.65	0.59	0.98	0.98	37.0	62.0	0.191	0.778	0.969	129	30.0	29.2	.....	.....
	4B		26.2	38.7	37.3	65.7	80.9	104.6	26.6	39.0	37.6	71.1	66.4	1.04	1.04	0.59	0.55	1.01	1.01	32.5	53.3	0.209	0.582	0.791		29.6	29.8	.....	.....
	5A		25.6	31.7	28.0	58.0	70.5	112.3	25.4	32.7	31.1	65.0	61.0	1.13	1.05	0.55	0.50	1.03	1.11	38.5	63.9	0.196	0.825	1.021	110	29.3	30.8	.....	.....
	5B		26.2	30.3	28.2	56.3	67.5	112.5	24.1	30.4	28.7	60.0	56.9	1.07	1.06	0.54	0.51	1.00	1.02	42.0	59.9	0.185	0.730	0.915		29.6	28.4	.....	.....
	6A		27.0	32.9	29.5	62.8	73.2	114.9	25.4	31.4	29.6	66.2	61.7	1.11	1.06	0.52	0.47	0.96	1.00	38.0	55.4	0.152	0.654	0.806	106	29.4	29.8	0.284	0.299
	6B		29.9	36.1	34.1	72.5	85.9	108.2	31.8	35.2	33.4	76.4	71.2	1.06	1.05	0.50	0.46	0.97	0.98	28.0^b	35.6	0.168	0.284	0.452		29.5	29.8	0.308	0.293
	7A		28.7	31.8	28.6	56.7	69.1	104.0	30.5	32.0	29.4	61.1	57.4	1.11	1.09	0.56	0.52	1.00	1.03	45.5	61.1	0.197	0.747	0.944	98	29.6	29.5	0.292	0.298
	7B		25.6	31.0	27.6	56.6	67.5	115.3	28.0	30.6	28.9	61.1	56.5	1.12	1.06	0.55	0.50	0.99	1.04	35.5	57.5	0.178	0.679	0.857		29.1	29.3	0.285	0.292
	8A	⅞-in. angle	31.6	37.8	35.6	61.9	73.6	127.9	39.9	40.6	39.4	71.2	65.9	1.06	1.03	0.61	0.57	1.07	1.10	39.0	65.6	0.173	0.896	1.069	120	29.4	29.7	0.288	0.303
	8B		29.4	33.6	29.4	57.2	71.7	106.0	28.8	33.6	31.4	61.1	56.9	1.14	1.07	0.59	0.55	1.00	1.07	34.5	55.8	0.225	0.593	0.818		29.6	29.5	0.301	0.293
	9A	¾-in. plate	23.7	31.2	28.8	58.1	68.5	117.9	24.2	31.1	28.3	61.1	56.3	1.08	1.10	0.54	0.51	1.00	0.98	41.0	62.4	0.161	0.815	0.976	124	29.4	29.3	0.286	0.295
	9B		23.7	31.2	28.3	58.5	68.9	107.2	22.5	31.3	28.7	65.0	61.7	1.10	1.09	0.53	0.48	1.00	1.01	39.0	54.7	0.162	0.630	0.792		29.2	29.4	0.307	0.295
	10A		32.4	35.0	31.8	63.1	78.1	136.8	30.0	35.0	31.1	66.2	62.0	1.10	1.13	0.55	0.53	1.00	0.98	39.5	59.9	0.212	0.700	0.912	122	29.6	29.5	0.306	0.301
	10B		23.7	36.2	31.2	63.7	76.4	117.7	30.0	33.8	31.2	71.2	65.9	1.16	1.08	0.57	0.47	0.93	1.00	37.0	55.4	0.181	0.626	0.807		29.3	29.4	0.299	0.296
	11A	1-in. plate	20.6	29.5	28.5	62.5	73.7	129.2	23.7	30.8	29.3	71.2	65.6	1.03	1.05	0.47	0.43	1.05	1.03	39.0	64.4	0.163	0.868	1.031	121	29.6	29.6	0.310	0.299
	11B		23.9	29.0	27.4	64.4	76.1	116.3	19.1	29.4	28.1	71.2	65.9	1.06	1.05	0.45	0.41	1.01	1.02	36.5	53.5	0.166	0.598	0.764		29.2	29.5	0.303	0.306
Avg. C.S.	With Roll "A"		28.1	34.1	31.4	60.2	73.3	117.6	28.4	34.1	31.9	65.0	60.7	1.085	1.070	0.565	0.525	1.001	1.014	40.2	62.0	0.196	0.774	0.970		29.57	29.55	0.294	0.299
	Cross Roll "B"		26.3	33.5	30.4	60.6	73.3	110.5	27.6	33.0	30.8	65.9	61.4	1.100	1.069	0.553	0.500	0.984	1.013	36.0	55.4	0.189	0.633	0.822	113	29.43	29.42	0.301	0.296
	All C.S.		27.2	33.8	30.9	60.4	73.3	114.0	28.0	33.5	31.3	65.4	61.1	1.093	1.070	0.559	0.513	0.993	1.014	38.1	58.7	0.193	0.703	0.896		29.50	29.48	0.297	0.298
Silicon Structural	12A	¾-in. plate	38.6	49.2	44.0	81.4	91.7	143.2	35.6	48.5	45.5	91.3	85.5	1.12	1.07	0.60	0.52	0.98	1.03	36.5	57.8	0.119	0.745	0.864	157	29.2	29.5	0.306	.....
	12B		35.1	48.7	44.5	81.5	94.1	124.5	35.6	46.9	44.4	91.3	85.7	1.09	1.06	0.60	0.51	0.96	1.00	31.5	49.3	0.144	0.535	0.679		29.0	29.3	0.295	.....
	13A		35.0	47.9	43.7	82.1	98.3	151.1	34.3	47.6	44.0	91.3	85.7	1.09	1.08	0.58	0.52	0.99	1.01	34.5	60.4	0.180	0.745	0.925	162	29.2	29.4	0.299	.....
	13B		23.7	43.4	43.1	81.4	95.1	128.8	34.3	46.8	44.6	91.3	85.7	1.01	1.05	0.53	0.51	1.08	1.03	32.5	45.8	0.155	0.457	0.612		29.3	29.6	0.286	.....
	14A		39.8	48.6	47.4	94.2	108.9	168.3	35.7	50.4	48.4	101.9	96.0	1.02	1.04	0.52	0.49	1.04	1.02	28.5	55.3	0.145	0.660	0.805	179	29.6	29.7	0.313	.....
	14B		42.4	48.0	46.5	90.1	112.4	142.9	36.8	48.8^a	None	106.5	100.7	1.03	....	0.53	0.46	1.02	....	16.5^b	45.1	0.167	0.482	0.649		29.8	29.5	0.302	.....
	15A		40.0	46.9	44.1	84.0	104.7	165.8	35.7	47.7	45.3	96.5	89.0	1.06	1.05	0.56	0.49	1.02	1.03	27.5	60.8	0.224	0.713	0.937		29.3	29.3	0.301	.....
	15B		27.5	41.2^a	None	77.9	92.9	124.4	27.9	40.1^a	None	81.2	77.1	....	....	0.53	0.49	0.97	....	30.5	45.4	0.175	0.430	0.605	161	29.5	29.3	0.301	.....
	15C		35.1	40.8	39.3	77.6	92.2	157.0	38.8	41.1	40.3	87.3	80.3	1.03	1.02	0.53	0.47	1.01	1.03	35.0	62.3	0.174	0.802	0.976		29.8	29.6	0.289	0.289
	16A		30.8	41.5	39.3	80.6	95.7	150.2	29.3	42.2	40.3	91.5	85.5	1.06	1.05	0.51	0.46	1.02	1.02	35.5	58.3	0.171	0.703	0.874	163	29.3	29.9	0.283	0.307
	16B		27.5	42.4	40.0	83.0	98.3	132.3	30.6	41.8	39.4	91.7	85.4	1.06	1.06	0.51	0.46	0.99	0.99	23.0^b	46.1	0.170	0.447	0.617		29.9	29.2	0.294	0.296
	17A		34.9	46.2	42.8	86.3	101.4	160.9	35.7	45.8	43.5	96.8	90.5	1.08	1.05	0.54	0.47	0.99	1.01	33.0	58.0	0.161	0.603	0.764	169	29.8	29.9	0.291	0.315
	17B		28.7	44.9	42.6	86.5	99.2	151.2	29.3	46.8	43.9	101.7	94.8	1.05	1.07	0.52	0.46	1.04	1.03	30.5	52.0	0.135	0.598	0.733		29.7	29.5	0.294	0.293
	18A	1-in. plate	31.1	45.4	40.9	79.8	94.0	159.7	39.4	43.4	40.8	91.6	84.4	1.11	1.06	0.57	0.47	0.96	1.00	34.0	62.9	0.163	0.828	0.991	158	29.5	29.4	0.292	0.301
	18B		26.1	42.1	37.7	76.5	89.8	128.2	29.3	41.5	38.4	86.6	75.0	1.12	1.08	0.55	0.48	0.99	1.02	28.5	46.8	0.160	0.471	0.631		29.7	29.2	0.297	0.301
Avg. S.S.	With Roll "A"		35.7	46.5	43.2	84.1	99.2	157.0	35.1	46.5	44.0	94.4	88.1	1.077	1.057	0.554	0.493	1.000	1.017	32.8	59.1	0.166	0.714	0.880		29.41	29.59	0.298	0.307
	Cross Roll "B"		21.1	44.4	42.4	82.4	97.4	133.2	32.0	44.7	42.1	92.9	86.3	1.060	1.064	0.539	0.481	1.007	1.014	27.6	47.2	0.158	0.488	0.646	164	29.55	29.37	0.296	0.297
	All S.S.		28.4	45.5	42.8	83.2	98.3	145.1	33.0	45.6	43.1	93.7	87.2	1.069	1.061	0.547	0.487	1.004	1.016	30.2	53.1	0.162	0.601	0.763		29.48	29.48	0.297	0.302

Low Alloy A-1	19A	¾-in. plate	28.7	44.8	40.8	77.0	90.7	172.0	40.7	45.0	42.0	86.5	80.9	1.09	1.07	0.58	0.52	1.00	1.03	37.0	68.8	0.166	1.000	1.166	163	29.6	29.6	0.303	0.295
	19B		36.2	43.6	40.5	75.9	89.9	135.1	38.2	43.8	40.8	86.5	80.6	1.08	1.07	0.57	0.51	1.00	1.01	33.0	52.0	0.170	0.564	0.734		29.7	29.9	0.309	0.284
	20A		26.2	44.9	40.9	75.1	89.4	163.5	30.0	44.8	41.5	86.5	80.0	1.10	1.08	0.60	0.52	1.00	1.01	37.0	68.4	0.173	0.978	1.151	149	29.8	29.2	0.299	0.294
	20B		23.7	43.0	39.8	74.1	88.3	131.0	26.7	42.3	40.9	86.5	80.4	1.08	1.04	0.58	0.49	0.98	1.03	33.5	51.1	0.175	0.549	0.724		29.8	29.9	0.304	0.305
L.A. A-2	21A	¾-in. plate	35.4	41.1	37.0	64.2	76.3	142.4	36.6	43.1	40.5	76.4	71.4	1.11	1.06	0.64	0.56	1.05	1.09	39.0	68.8	0.175	0.991	1.166	131	29.6	29.8	0.292	0.305
	21B		36.0	42.5	38.5	63.6	77.4	143.1	35.1	40.5	37.8	71.2	66.2	1.09	1.07	0.67	0.57	0.95	0.98	43.5	71.4	0.195	1.056	1.251		29.6	29.6	0.299	0.307
Low Alloy A-3	22A	¾-in. plate	41.2	52.5	48.5	75.4	88.1	158.8	39.4	51.9	49.5	81.3	76.3	1.08	1.05	0.70	0.64	0.99	1.02	37.5	69.5	0.156	1.016	1.172	149	29.4	29.8	0.277	0.291
	22B		40.0	50.6	47.2	74.5	88.1	154.2	41.2	52.3	49.2	86.5	8 .3	1.07	1.06	0.68	0.60	1.03	1.04	32.0	54.6	0.168	0.622	0.790		29.6	29.8	0.295	0.304
	23A		36.2	53.1	46.5	70.9	87.0	151.3	39.5	51.0	47.5	81.6	75.3	1.14	1.07	0.75	0.62	0.96	1.02	40.0	70.1	0.204	1.005	1.209	141	29.7	29.6	0.297	0.292
	23B		33.7	47.4	42.5	69.0	86.3	132.5	43.2	47.1	44.1	76.4	71.1	1.11	1.07	0.69	0.62	0.99	1.04	30.0	58.6	0.222	0.659	0.881		29.8	29.8	0.290	0.292
	24A	1-in. plate	31.2	46.1	43.9	72.0	85.2	163.7	39.2	47.4	44.9	86.5	81.1	1.05	1.05	0.64	0.55	1.03	1.02	37.0	72.3	0.168	1.122	1.290	154	29.6	29.8	0.299	0.290
	24B		32.4	46.4	44.1	71.9	86.6	166.9	33.1	47.3	45.0	81.4	75.9	1.05	1.05	0.65	0.58	1.02	1.02	34.0	61.1	0.187	0.758	0.945		29.5	29.7	0.300	0.299
Low Alloy A-4	28A	¾-in. plate	37.4	48.6	41.8	68.9	81.9	159.3	34.4	45.1	42.0	75.4	71.1	1.16	1.07	0.71	0.60	0.93	1.00	41.0	71.6	0.174	1.087	1.261	140	29.9	29.5	0.296	0.292
	28B		38.7	45.5	42.1	68.9	83.4	124.4	37.7	46.1	42.8	81.4	75.3	1.08	1.08	0.66	0.57	1.01	1.02	36.0	54.7	0.191	0.600	0.791		29.6	29.6	0.291	0.295
	29A		25.9	42.4	41.4	76.5	90.2	167.7	30.5	43.4	41.6	91.6	83.5	1.02	1.04	0.55	0.47	1.02	1.00	34.5	69.1	0.166	1.007	1.173	155	29.3	29.8	0.289	0.293
	29B		28.6	41.9	39.6	74.8	88.8	134.3	34.6	43.8	41.8	91.8	84.1	1.06	1.05	0.56	0.48	1.05	1.05	34.5	55.4	0.172	0.634	0.806		29.5	29.5	0.290	0.294
L.A. A-5	30A	¾-in. plate	26.4	43.5	41.2	74.1	87.6	147.1	29.3	45.5	42.9	86.5	79.8	1.06	1.06	0.59	0.53	1.05	1.04	35.5	63.5	0.166	0.842	1.008	147	29.5	29.8	0.294	0.292
	30B		32.9	43.4	41.4	74.2	87.6	131.5	32.1	45.1	42.9	81.5	76.8	1.05	1.05	0.58	0.55	1.04	1.03	30.0	53.9	0.166	1.070	1.236		29.4	29.3	0.301	0.300
L.A. A-6	31A	¾-in. plate	27.5	49.0	46.9	82.1	97.0	146.2	37.0	50.9	48.1	96.9	90.0	1.04	1.06	0.60	0.53	1.04	1.02	33.0	57.3	0.165	0.686	0.851	158	29.6	29.5	0.298	0.305
	31B		31.4	51.0	48.9	87.4	99.8	150.7	42.2	51.2	49.0	96.6	89.5	1.04	1.04	0.58	0.53	1.00	1.00	30.0	56.9	0.181	0.660	0.841		29.3	29.3	0.304	0.303
Avg. L.A. "A"	With Roll "A"		31.6	46.6	42.9	73.6	87.3	157.2	35.7	46.8	44.0	84.9	78.9	1.087	1.063	0.633	0.554	1.005	1.025	37.1	67.9	0.171	0.973	1.145		29.60	29.64	0.294	0.295
	Cross Roll "B"		33.4	45.5	42.5	73.4	87.7	140.4	36.4	46.0	43.4	84.0	78.0	1.072	1.058	0.620	0.543	1.007	1.022	33.7	57.0	0.183	0.717	0.900	149	29.58	29.64	0.298	0.298
	All L. A. "A"		32.5	46.1	42.7	73.5	87.5	148.8	36.0	46.4	43.7	84.5	78.4	1.080	1.061	0.627	0.549	1.006	1.024	35.4	62.5	0.177	0.845	1.022		29.59	29.64	0.296	0.297
Low Alloy B-1	25A	¾-in. plate	35.0	52.5	47.1	81.5	96.4	179.7	34.4	50.9	48.0	91.6	84.6	1.11	1.06	0.64	0.56	0.97	1.02	35.5	71.4	0.168	1.082	1.250	166	29.6	29.8	0.281	0.302
	25B		31.7	51.2	47.2	81.7	97.1	170.5	39.5	52.7	48.8	91.7	85.7	1.08	1.08	0.63	0.57	1.03	1.03	33.5	67.5	0.172	0.950	1.122		29.7	29.7	0.290	0.299
	26A		36.4	54.0	52.0	90.1	105.0	183.9	39.4	54.0	52.5	101.8	96.0	1.04	1.03	0.60	0.53	1.00	1.01	30.5	68.9	0.153	0.930	1.083	170	29.6	29.8	0.298	0.290
	26B		36.2	54.6	52.9	90.9	104.5	177.0	42.0	54.7	53.1	101.8	95.5	1.03	1.03	0.60	0.54	1.00	1.00	29.0	63.8	0.139	0.875	1.014		29.7	29.6	0.294	0.306
	27A		48.1	53.1	50.4	78.4	93.2	186.3	39.4	53.9	50.9	91.6	84.7	1.05	1.06	0.68	0.59	1.01	1.01	37.5	75.5	0.173	1.232	1.405	170	29.8	30.0	0.295	0.296
	27B		38.7	53.7	49.2	78.6	91.4	168.9	43.3	52.8	50.0	86.5	80.5	1.09	1.06	0.68	0.61	0.98	1.02	37.0	70.1	0.151	1.056	1.207		29.7	30.1	0.298	0.295
Low Alloy B-2	32A	¾-in. plate	41.2	51.7	47.0	66.1	79.4	131.1	51.7	51.7	47.3	76.3	71.2	1.10	1.09	0.78	0.68	1.00	1.01	35.5	65.0	0.182	0.866	1.048	121	29.5	29.1	0.306	0.298
	32B		45.4	51.1	46.6	65.6	78.8	135.0	52.2	52.2	47.4	76.3	71.5	1.10	1.10	0.78	0.68	1.02	1.01	33.0	61.4	0.183	0.768	0.951		29.2	29.3	0.291	0.297
	33A		33.7	57.2	54.4	85.0	101.6	163.6	47.7	59.6	56.9	96.8	90.7	1.05	1.05	0.67	0.62	1.04	1.04	30.5	62.3	0.179	0.797	0.976	178	29.5	29.3	0.296	0.287
	33B		33.6	57.9	56.1	86.0	101.6	147.9	45.8	60.6	57.5	96.7	91.1	1.03	1.05	0.67	0.63	1.05	1.02	27.5	47.9	0.166	0.484	0.650		29.3	29.4	0.306	0.304
Avg. L.A. "B"	With Roll "A"		38.9	53.7	50.2	80.2	95.1	168.9	42.5	54.0	51.1	91.6	85.4	1.070	1.058	0.669	0.592	1.004	1.018	33.9	68.6	0.171	0.981	1.152		29.60	29.60	0.295	0.295
	Cross Roll "B"		37.1	53.7	50.4	80.6	94.7	159.9	44.6	54.6	51.4	90.6	84.8	1.066	1.064	0.667	0.603	1.016	1.019	32.0	62.1	0.162	0.827	0.989	161	29.52	29.62	0.296	0.300
	All L. A. "B"		38.0	53.7	50.3	80.4	94.9	164.4	43.5	54.3	51.2	91.1	85.1	1.068	1.061	0.668	0.598	1.010	1.019	33.0	65.4	0.167	0.904	1.071		29.56	29.61	0.296	0.298
Total Avg.	With Roll "A"		....	....	....	....	....	....	....	....	....	....	....	1.082	1.063	0.599	0.537	1.002	1.019	....	....	0.178	0.853	1.031		29.55	29.59	0.295	0.298
	Cross Roll "B"		....	....	....	....	....	....	....	....	....	....	....	1.078	1.064	0.588	0.525	1.001	1.017	....	....	0.177	0.657	0.834	...	29.51	29.51	0.298	0.298
	All Steels		....	....	....	....	....	....	....	....	....	....	....	1.080	1.064	0.594	0.531	1.002	1.018	....	....	0.178	0.755	0.933		29.53	29.55	0.297	0.298
Alum. Alloy 27S-T	34A	1-in. plate	18.7	40.5[a]	....	59.5	70.5	70.6	19.1	40.0[a]	....	58.6	56.3	.....	.....	0.68[a]	0.68[a]	0.99	.....	17.5	22.9	0.170	0.037	0.207		10.7	10.9	0.345	0.345
	34B		28.6	40.3[a]	....	58.7	66.0	68.0	26.8	41.5[a]	....	61.1	58.1	.....	.....	0.67[a]	0.68[a]	1.03	.....	13.5	13.7	0.118	0.029	0.147	...	10.9	10.9	0.336	0.356
	35A	1-in. rod	36.4	53.5[a]	....	68.3	74.3	85.5	47.8	55.4[a]	....	73.7	69.1	.....	.....	0.78[a]	0.75[a]	1.03	.....	16.5	32.6	0.085	0.310	0.395		10.6	10.7	0.350	0.354

[a] Yield strength at 0.002 offset.

[b] Broke near punch mark.

at right angles to each other in order to secure freedom of motion in all directions. These hardened knife-edges fitted into hardened bearing plates with slight V's cut into them to produce self-alignment (see Fig. 1). The frictional resistance to rotation in this type of device was very low, and by adjusting the apparatus to compensate for errors in the machining of specimens it was possible to obtain a high degree of load axiality. The axiality in each test was checked by tensometer readings taken at 120-deg. intervals around the axis of the specimen, as shown in Fig. 1, loading the specimen for this purpose to a stress of approximately 5000 psi.

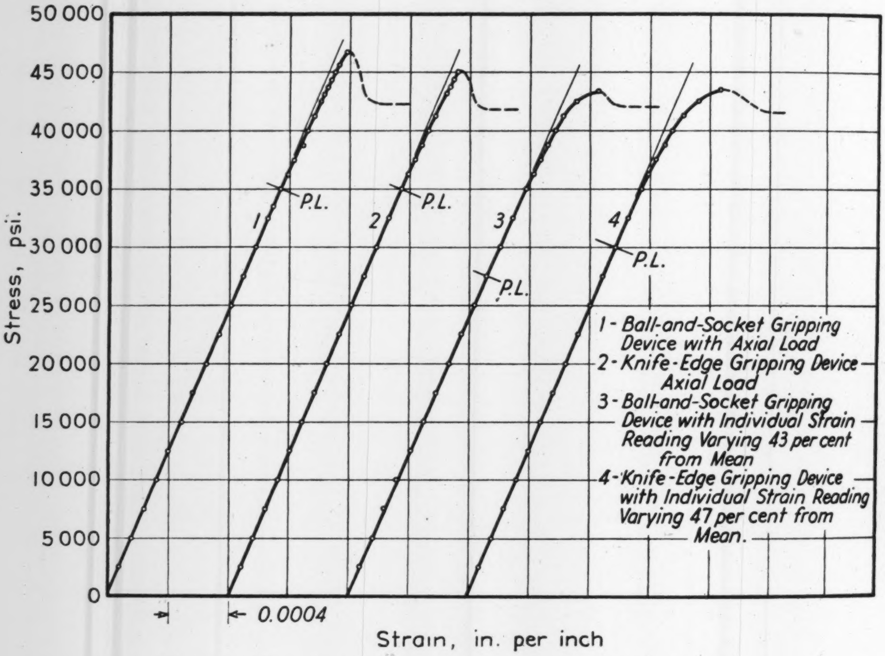

FIG. 2.—Stress-Strain Curves for Different Test Conditions.

Several tests were made to compare results obtained by the ordinary ball-and-socket device with results obtained by the knife-edge device. These tests were run on one sample of steel and the results are shown in Fig. 2. Eccentricities were introduced, in the case of the knife-edge rig, by rotation of the specimen with respect to the gripping device. In the case of the ball-and-socket loading device, eccentricities were introduced by displacing the wedges in such a manner as to cause the ball to rest upon an uneven surface. Figure 2 indicates that the type of loading device seems to have little effect upon either the shape of the stress-strain curve or the yield point of the material as long as the axiality can be controlled. Nonaxiality of load produced a marked curvature in the upper part of the stress-strain diagram. These tests indicate that if care is taken in aligning the split jaws good results can be expected with the ball-and-socket loading device. The knife-edge device was used for all of the tension tests, however, because it offered the most convenient control for axiality initially and throughout each test.

For compression tests of metals, the A.S.T.M. Tentative Methods of Compression Testing of Metallic Materials (E 9 - 33 T)[11] recommend that tests be made with a fixed base and a spherical bearing block for the upper bearing surface. The advice of other investigators indicated that with this type of testing device it would be rather difficult to secure precise control throughout the range of the test.

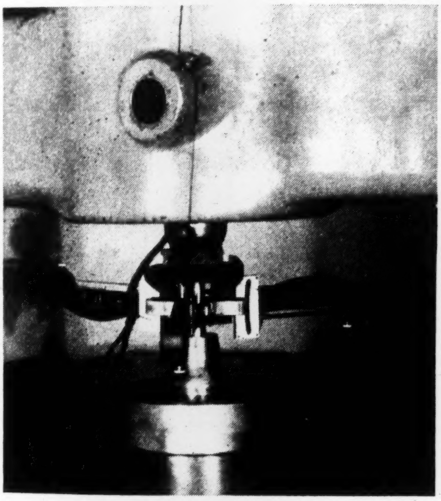

Fig. 3.—Compression Test Setup, with Tensometers and Lateral Strain Gage in Place.

Fixed heads with parallel faces are thought to provide the best testing device. Accordingly two bearing blocks, fitted with hardened steel faces, were machined so that one of them could be firmly fastened to the head of the testing machine. The other bearing block was placed on the table of the testing machine (see Fig. 3). The specimen was inserted, carefully centered, and a small load applied to determine whether the loading was axial, as indicated by the readings of the three extensometers. If the loading was not axial cigarette paper was used to shim up the face of the $5\frac{1}{2}$-in. diameter base of the bottom bearing block. In no case was it necessary to use more than three or four thicknesses of cigarette paper (about 0.003 to 0.004 in.) in order to secure good agreement between the readings on the extensometers. It is important to note that in order to eliminate bending while testing the specimen in compression, the bearing faces of the blocks must remain parallel to each other throughout the range of the test. The parallelism of the blocks was checked by placing a 10-sec. level bubble on the movable table of the testing machine. It was found that the maximum angular movement during the range of the test amounted to about two divisions of the level bubble, which movement can be expressed as approximately 0.00001 in. per inch. Such a small change in parallelism could not cause measurable strain differences.

The shape and size of the compression test specimens were selected on the basis of several factors. A specimen $\frac{1}{2}$ in. in diameter was decided upon because it would be approximately the same size as the tension specimen. A 1-in. length of uniform stress condition was needed to measure properly the elastic constants, since the extensometers had a 1-in. gage length. Nadai[4] has shown that specimens in compression have a region at each end of comparatively little distortion bounded by a cone approximately one half the diameter in height. In order that the gage length be free of these end effects, the length of a $\frac{1}{2}$-in. diameter specimen would need to be $1\frac{1}{2}$ in. or more. A series of tests was run using various ratios of length to diameter (*L*/*D*), and the resulting upper yield point stresses are plotted in

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[11] 1939 Book of A.S.T.M. Standards, Part I, p. 1230.

Fig. 4. Ultimate failure on specimens with *L*/*D* ratios greater than 2 occurred as a plastic buckling failure. However, the lowest ultimate load for an *L*/*D* ratio of 6 was more than $1\frac{1}{2}$ times as great as the yield point load. Buckling failure was therefore not a factor in the variation of the yield points, and Fig. 4 shows practically no variation in the upper yield point for *L*/*D* ratios between 4 and 6. With these results as a basis, an *L*/*D* ratio of 4 was adopted, and a specimen $\frac{1}{2}$ by 2 in. in length was used for all compression tests. In the special alternating tension and compression tests to observe the Bauschinger effect a standard tension test specimen was used, with the ends faced parallel in the lathe for bearing in the compression cycles.

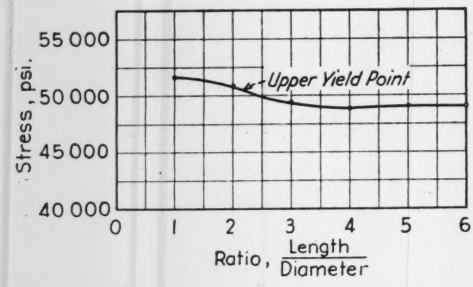

FIG. 4.—Upper Yield Point for Compression Tests with Different Ratios of $\frac{L}{D}$.

To determine whether end conditions of the specimen had anything to do with yield point or stress-strain characteristics, several specimens had their ends surface-ground, while another group had the ends faced on a lathe. No difference due to the difference in machining was observed. The side surface condition in both the tension and compression test specimens was that left by a finishing cut with a lathe tool followed by light polishing with an emery cloth.

Each test required at least thirty minutes to complete in the elastic range. The rate of loading and rate of deformation were slow at all times and not great enough to be considered as affecting either the yield point or the shape of the stress-strain curve. The rate of deformation was increased slightly in the plastic range but at no time did the speed of the head exceed 0.05 in. per minute.

The measurements for the modulus of elasticity were made by means of three Huggenberger Type A tensometers mounted 120 deg. apart around the specimen as shown in Figs. 1 and 3. This mounting was used to facilitate detection and adjustment of nonaxiality of loading. The Huggenberger tensometer is sensitive to a strain of 0.00001 in. per inch. The tensometers were used up to the yield point and the maximum deviation of any single reading was rarely greater than 3 per cent of the average of the three tensometers.

An electrical lateral strain gage developed by Massachusetts Institute of Technology[12] was used to measure lateral strains and thereby determine Poisson's ratio. This gage is sensitive to strains of 0.000002 in. per inch.

All of the strain-measuring devices used in these tests were calibrated by means of an interferometer calibrating instrument, using the 5461 Ångström line of the mercury arc as the basis of comparison.

The changes in diameter of the specimen beyond the yield point (after removal of strain gages) were measured by means of two 0.001-in. dial gages. One of the gages projected through a V-block while the other was mounted in an opposed position. The whole assembly could be moved over the length of the specimen and changes in diameter determined while the deformation was taking place.

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[12] Manufactured under license from the Baldwin-Southwark Corp.

In order to illustrate how the previous strain history of the metal alters its stress-strain characteristics, a series of tests was run involving cyclic loadings of both steel and aluminum. Much more extensive investigations on this subject have been carried on by other investigators; for example, Bauschinger,[13] Muir,[14] and more recently Templin and Sturm.[15]

The result of cyclic loading of a carbon structural steel sample (test specimen 9B) is shown in Fig. 5. The cycle of curves on the left illustrates (1) initial tensile load just into the lower yield point range; (2) removal of tensile load; (3) compression load, with a considerable decrease in the proportional limit as compared with a previously unstrained specimen (1, on right); (4) removal of compressive load; (5) tensile load, with low proportional limit; (6) removal of tensile load; and, finally (7) restoration of initial tensile characteristics, showing hysteresis loop. Note that the phenomena of upper and lower yield points have been maintained in loadings 3, 5, and 7. During loadings 3 and 5, although the shape of the stress-strain curve has been altered markedly, the yield points are not reduced, nor are these less than the yield strength as based on a 0.002 offset.

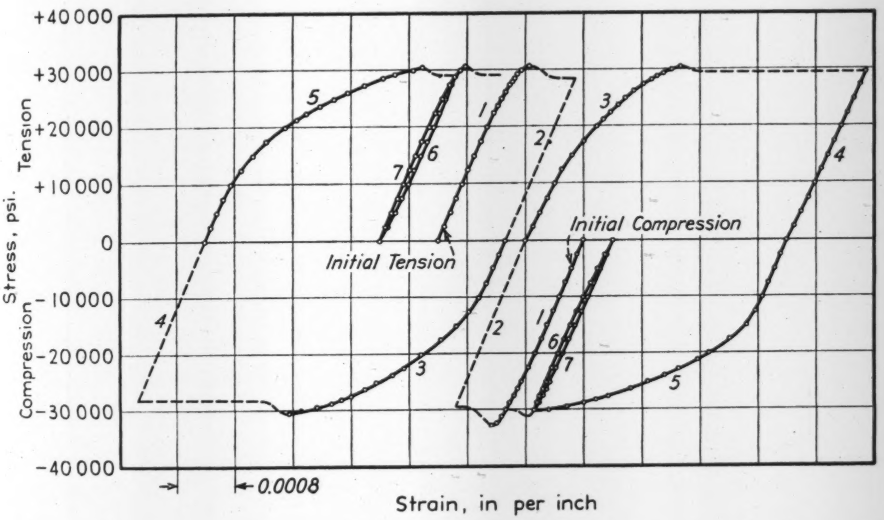

Fig. 5.—Cyclic Stress-Strain Curves in Tension and Compression for Structural Steel Sample 9B.

The right side of Fig. 5 exhibits almost the same characteristics for the complete reverse of the order on the left, starting in this case with initial compression.

Figure 6 shows two similar cycles of loading for specimens taken from 1-in. diameter rod of aluminum alloy 27S-T (test specimen No. 35). The general shape of the curves after previous strain in the reverse direction is not changed as much for the aluminum alloy as for the structural steel. The use of an 0.002 offset as the arbitrary definition for yield strength of the aluminum alloy, however, would lead to the conclusion that the yield strength is lowered after previous stress in the reverse direction up to or slightly beyond the yield strength.

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[13] J. Bauschinger, "Die Veränderungen der Elasticitätsgrenze," *Mitteilungen*, Mechanical Testing Lab. (München) (1886).

[14] James Muir, "On the Recovery of Iron from Overstrain," *Philosophical Transactions*, Royal Soc. (London), Vol. 193, pp. 1-45 (1900).

[15] R. L. Templin and R. G. Sturm, "Some Stress-Strain Studies of Metals," *Journal of the Aeronautical Sciences*, Vol. 7, No. 5, March, 1940, pp. 189-198.

In Fig. 5, the amount of overstrain in the lower yield point range could not be measured exactly but was approximately ½ per cent. In the case of the aluminum alloy the overstrain corresponded approximately to that obtained at the yield strength.

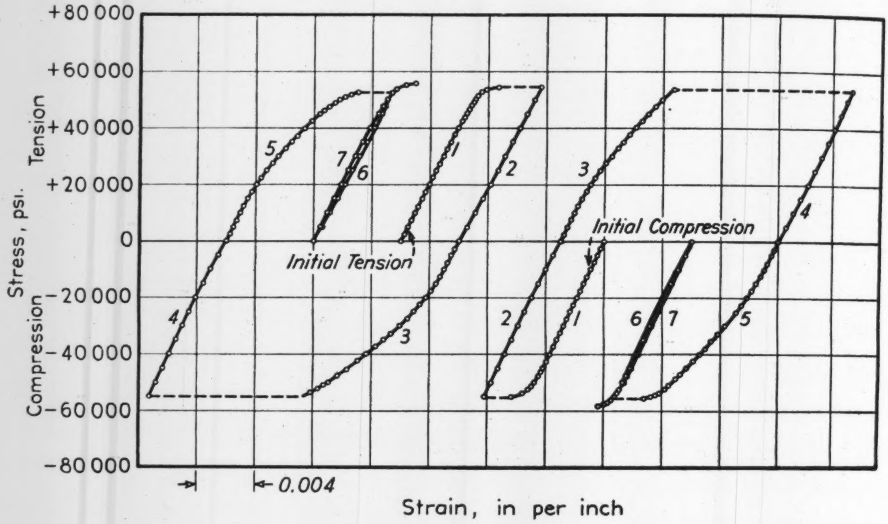

FIG. 6.—Cyclic Stress-Strain Curves in Tension and Compression for Structural Aluminum Alloy 27S-T Sample No. 35A.

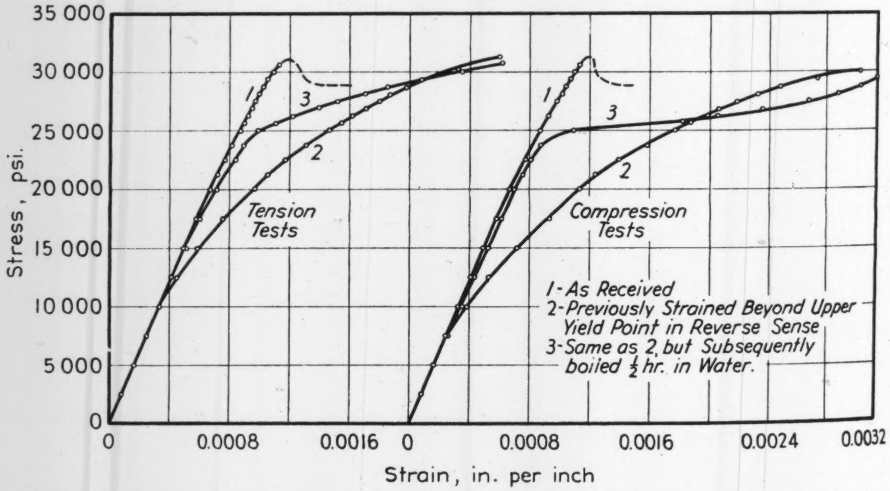

FIG. 7.—Effect of Prior Overstrain in Reverse Sense on Structural Steel Sample 9B and Partial Elastic Recovery by Boiling in Water.

Figure 7 illustrates the phenomena of elastic recovery, observed by James Muir[14] in 1900. On the left, (1) is the stress-strain diagram in tension for the undisturbed structural steel, (2) represents the tension test after previous compression just into the lower yield point range, and (3) is the same as (2) except for subsequent boiling in water for $\frac{1}{2}$ hr. The three curves on the right illustrate compression test results for the reversed conditions. The curves (3) in each case show the partial elastic recovery produced by boiling in water for a short period. Similar recovery is reported by Muir and others for specimens left for several days at ordinary room temperature.

TEST RESULTS

The general test results are summarized in Table II, the first three columns of which classify the material used in each test. The steels are classified in the first column into four general groups: carbon structural, silicon structural, low alloys A, and low alloys B. The low-alloy steels are further classified into subgroups according to different manufacturer's classifications. Each subgroup of low alloy A has some samples with upper yield points between 40,000 and 50,000 psi., whereas no single sample in low-alloy group B has an upper yield point below 50,000 psi. This classification of the low alloys is purely arbitrary and is for the purpose of comparing averages of the test results.

In the second column of Table II, the numerals refer to the different specimens and the letters A or B indicate tests with and at right angles to the direction of rolling, respectively. Test 15C is a special test at 45 deg. to the direction of rolling and is not included in the averages. The third column gives the source of the material.

Six columns under the heading Tension Test and five columns under the heading Compression Test present unit stresses at the proportional limit (σ_P), the upper yield point (σ_{UY}), the lower yield point (σ_{LY}), the nominal maximum (σ_{NM}), the true maximum (σ_{TM}), and the true breaking stress (σ_{TB}). In the case of the compression tests there is no true breaking stress and the values of maximum stress represent the stress when the test was stopped and at a more or less arbitrary load during initial plastic buckling. In three tests of silicon steels and all tests of aluminum alloy no lower yield points were observed and the yield strength of 0.002 per cent offset is reported under the column for (σ_{UY}).

The presentation of unit stresses is followed in Table II by six columns giving significant stress ratios and five columns presenting the usual ductility criteria together with the "true" indices proposed by MacGregor.[3] The last four columns give the experimental values of Young's modulus of elasticity and Poisson's ratio, determined both in tension and compression.

Table III presents the ratios between

TABLE III.—RATIOS BETWEEN RESULTS WITH DIRECTION OF ROLLING AND THOSE NORMAL TO DIRECTION OF ROLLING FOR AVERAGE PHYSICAL PROPERTIES SHOWING DEFINITE DIRECTIONAL EFFECT.

Steel Classification	Tension		Compression	Elongation in 2 in., per cent	Reduction of Area, per cent	True Ductility Indices		
	σ_P	σ_{TB}	σ_P			Uniform	Necking	Total
Carbon structural	1.069	1.064	1.035	1.117	1.119	1.037	1.223	1.180
Silicon structural	1.694	1.179	1.041	1.189	1.251	1.051	1.463	1.362
Low alloys A	0.948	1.120	1.019	1.101	1.193	0.934	1.357	1.272
Low alloys B	1.047	1.056	0.989	1.059	1.105	1.055	1.186	1.165
Weighted average	1.162	1.104	1.017	1.119	1.167	1.011	1.309	1.244

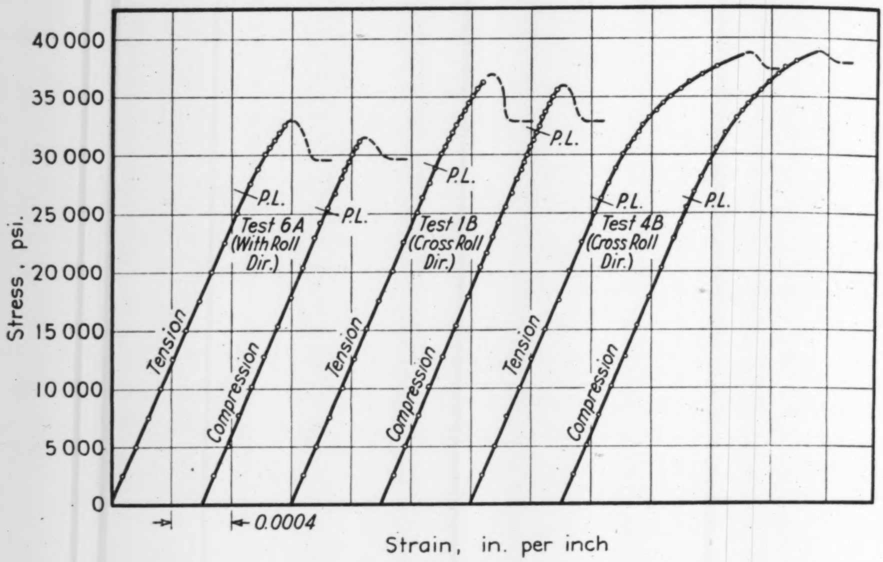

FIG. 8.—Typical Stress-Strain Curves in Tension and Compression for Tests of Carbon Structural Steel.

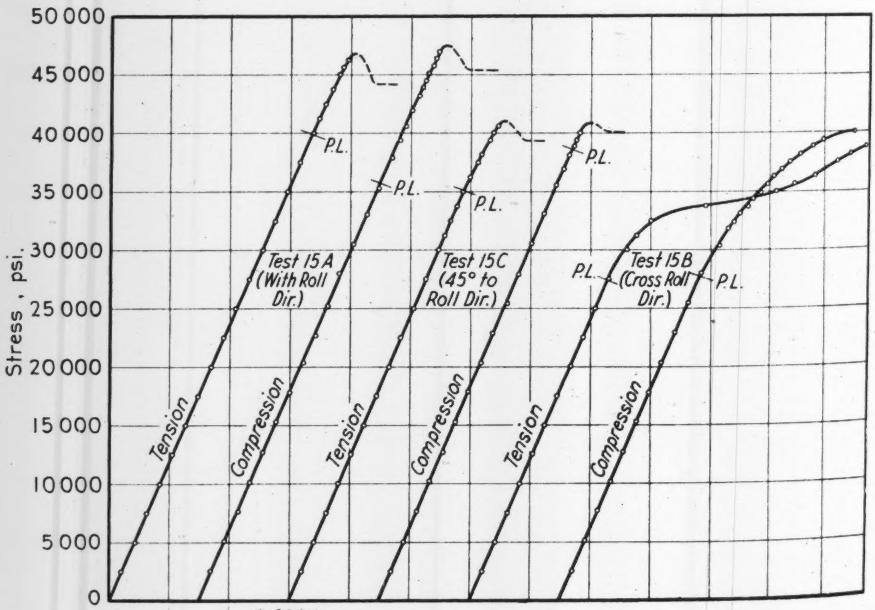

FIG. 9.—Stress-Strain Curves of a Silicon Structural Steel with Marked Directional Properties.

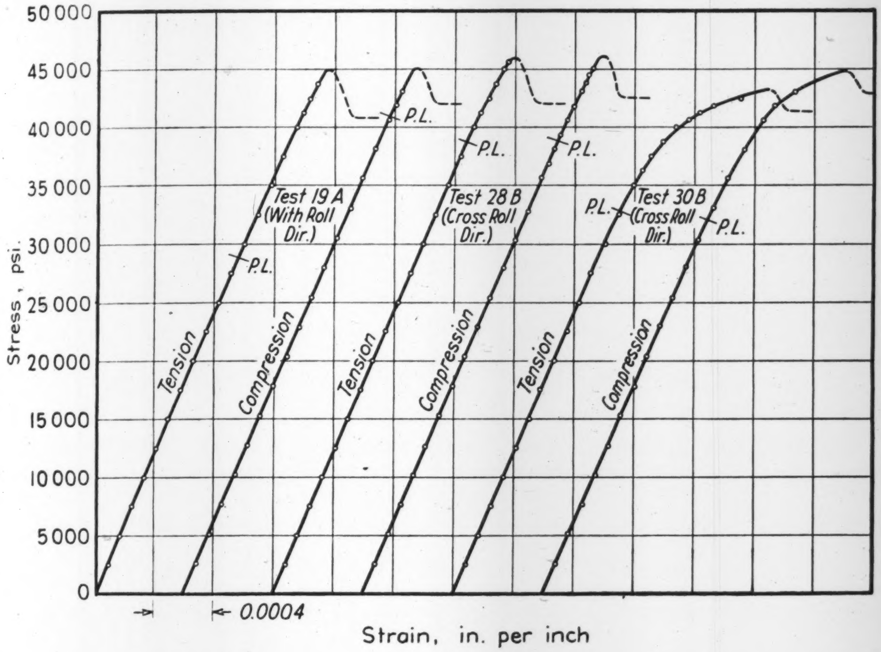

FIG. 10.—Stress-Strain Curves of Low Alloy Steels in Tension and Compression.

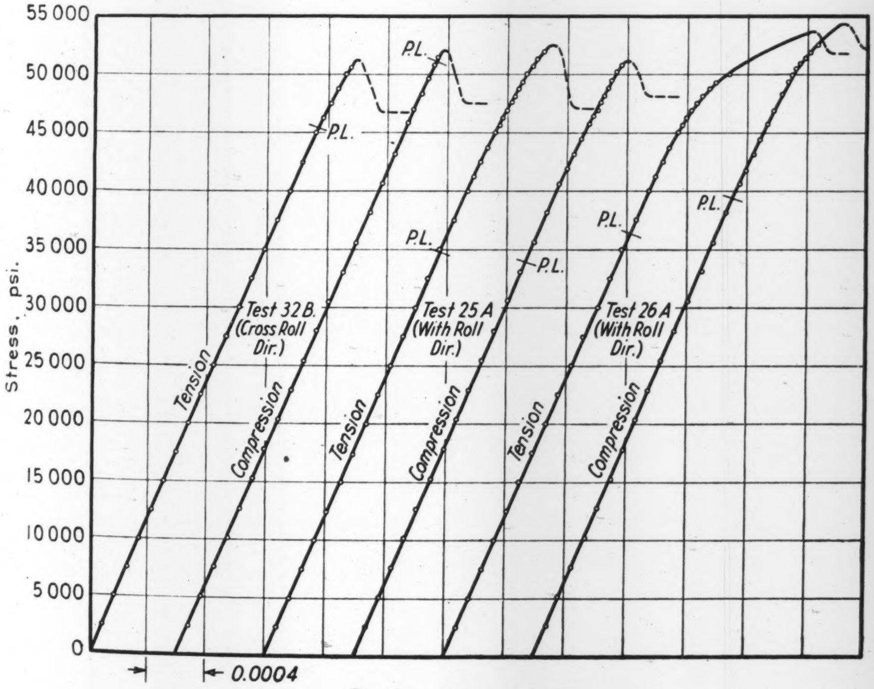

FIG. 11.—Stress-Strain Curves of Low Alloy Steels in Tension and Compression.

results with and across the direction of rolling for the group averages in Table II.

Figures 8, 9, 10, and 11 present some of the stress-strain curves in tension and compression for each of the groups of steels tested. In each of the figures are presented examples of (1) a test showing the most linear behavior up to the yield point, (2) a typical test, and (3) a test showing the least linear behavior. In Fig. 9, for silicon structural steel, the three examples of different behavior are all from the same sample but each test is oriented differently with respect to the direction of rolling.

DISCUSSION OF TEST RESULTS

Proportional Limit and Shape of Stress-Strain Diagram Below the Yield Point:

The influences of axiality of test load and previous strain history upon the proportional limit have been illustrated by special tests (see Figs. 2, 5, 6, and 7). The influence of stress raisers due to surface roughness or shoulder fillets in reducing both the proportional limit and yield points was illustrated by Docherty and Thorne.[10]

The average ratios between proportional limit and upper yield point for the steels tested are:

	With Direction of Rolling	Across Direction of Rolling	Average
Carbon structural steel	0.82	0.78	0.80
Silicon structural steel	0.77	0.48	0.62
Low alloys in group A	0.68	0.73	0.70
Low alloys in group B	0.72	0.69	0.71

In relation to the yield point, carbon structural steel had relatively the highest proportional limit and silicon structural steel the lowest. Furthermore, the silicon structural steel showed a marked lowering of the proportional limit in tension when tested at right angles to the direction of rolling.

The shape of the stress-strain curve above the proportional limit is important in determining critical buckling stresses in the design of plate elements in compression. The typical and extreme stress-strain curves in Figs. 8, 9, 10, and 11 provide some information along these lines but it should be kept in mind that the previous rolling mill treatment undergone by the material affects the results. All of the material tested was $\frac{3}{4}$ in. thick or more. Rolling the same material to smaller thicknesses might alter its stress-strain characteristics.

Upper and Lower Yield Point:

The upper yield point for any particular steel depends upon the previous strain history, the rate of loading, the presence of local stress raisers such as surface roughness or shoulder fillets, the axiality of test load (see Fig. 2), and the cross-sectional shape of the specimen. Higher upper yield points would have been noted if a larger radius had been used for the shoulder fillets.[5] It should also be noted that higher upper yield points would have resulted if the speed of test had been as great as that allowed by A.S.T.M. Standards.[16] The speed of test was so slow in these tests that it could not be considered an influencing factor.

The lower yield point is considered by some to be a more stable criterion than the upper and is affected principally by rate of strain and the previous strain history of the material. It is interesting to note that if the arbitrary offset criterion for yield strength were applied to a steel with a drop-of-the-beam yield point the lower yield point would correspond to the yield strength.

The ratio between upper and lower yield point is tabulated for each test in Table II. The upper yield point averaged 7.4 per cent higher than the lower yield point for all of the steels tested and this percentage did not vary greatly for the different steels or for the tests with or across the direction of rolling.

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[16] Joseph Winlock and Ralph W. E. Leiter, "Some Factors Affecting the Plastic Deformation of Sheet and Strip Steel," *Transactions*, Am. Soc. Metals, Vol. XXV, No. 1, March, 1937, pp. 163–185.

The ratio between upper yield point stress and nominal maximum stress is sometimes limited in structural specifications. Individual values of this ratio are also given in Table II, the ratios in the tension test having more significance than those in the compression test. The averages of this ratio were about the same for carbon structural and silicon structural steel and were slightly higher for the low-alloy steels.

Comparison between the yield points in tension and compression are particularly significant because they indicate to what extent the tension test may be relied upon to provide information regarding the compression stress-strain characteristics of the material. Considerable difference between the compression and tension stress-strain characteristics have been reported for some alloys used in aircraft construction.[17]

The structural and low-alloy steels tested in this program showed practically no difference between compression and tension stress-strain character istics through the upper and lower yield point range, as based on group averages for any particular direction of rolling. The grand average of all tests showed the upper yield point to be only 0.2 per cent higher in compression than in tension and the lower yield point to be 1.8 per cent higher. These differences are smaller than some of the other factors which may affect the test results.

Stresses at the Maximum and Breaking Loads:

Nominal and true stresses are reported in Table II for each test at the maximum and breaking loads. The maximum load in the tension test indicates the beginning of local necking but in the compression test signifies the load at plastic buckling. The true stress at the breaking load is reported for each tension test and averages higher in the direction of rolling than normal to it for all the steels tested. This is a result of the smaller reduction of area exhibited across the direction of rolling. The nominal and true stresses at maximum load were not influenced to any significant degree by the direction of rolling.

Ductility Criteria:

Table II presents the usual measures of ductility, that is, percentage elongation in 2 in. and percentage reduction of area. Both of these criteria indicate, for the averages of all steels tested, greater ductility with the direction of rolling than across.

The ductility indices proposed by MacGregor[3] are also tabulated in Table II. These "true" indices of ductility give a more complete picture of the ductility qualities than do the usual criteria. The only information required to calculate the "true" indices in addition to that obtained in the standard tension test are the measurements of the diameter of the specimen at maximum load and a recording of the load at fracture.

The uniform "true" ductility index for the steels tested in this program shows practically the same average value with and across the direction of rolling. The "necking" or "nonuniform" "true" index of ductility, on the other hand, is consistently higher for tests in the direction of rolling, with the greatest directional effect in the case of the silicon structural steel.

The individual tests of the steels show a fairly consistent relation between

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[17] William R. Osgood, "Column Strength of Tubes," *Report No. 615*, Nat. Advisory Committee for Aeronautics (1938).

"uniform" ductility and "necking" ductility, with the "necking" usually amounting to from three to six times the "uniform." The two specimens of structural aluminum alloy 27S-T show interesting individual behavior with respect to the "true" ductility indices. Specimens Nos. 34A and 34B show between four and five times more uniform ductility than necking ductility, both with and across the direction of rolling. The surface of these samples showed a stringy texture after elongation. Aluminum alloy No. 35, in contrast to Nos. 34A and 34B, exhibited nearly four times more necking ductility than uniform ductility, and the elongated material maintained a smooth surface appearance. Just what significance there is to these contrary types of behavior is open to question, but it may be important to note that the percentage elongation values completely mask these differences which are brought out so clearly by the "true" indices of ductility. An effort was made to get more samples of structural aluminum alloy but the defense situation had developed in the meantime and this was not possible.

Brinell Hardness Number:

The Brinell hardness numbers are based on impressions on the rolled surface after rough polishing. The approximate correlation of nominal maximum tensile strength in pounds per square inch with Brinell number (B) for all steels was

$$\sigma_{NM} = 11{,}500 + 428\ B.$$

The agreement in 32 out of 33 tests was within ±10 per cent. The relation $\sigma_{NM} = 500\ B$ agreed just as well for the silicon and low-alloy steels but gave in general too low values for the carbon structural steels.

Elastic Constants:

The determination of the modulus of elasticity and Poisson's ratio resulted in remarkably consistent values. It is believed that this is due in part to the simultaneous use of three tensometers during each test and to the extreme care used in obtaining axial loading.

The average values for all steels of $E = 29{,}540{,}000$ psi. and $\nu = 0.297$ may be used to calculate the shearing modulus, G.

$$G = \frac{E}{2(1 + \nu)} = 11{,}390{,}000 \text{ psi.}$$

The directional properties of the material had no apparent influence on either the modulus of elasticity or Poisson's ratio.

Directional Properties in the Plane of Plate:

The properties of the steels with respect to direction of rolling showed variations of group averages of more than a few per cent only in the case of the proportional limit, true breaking stress, and some of the ductility indices (see Table III). The index of "uniform" ductility showed no appreciable directional properties. In view of these facts it may be concluded that structural steels, on the average, exhibit very little directional properties in the plane of the plate up to the maximum test load in so far as the tension test is a criterion. Individual exceptions to this conclusion were noted, as is shown by Fig. 9 for a silicon structural steel. In this case the yield point, as well as the proportional limit, was affected. In general, the silicon structural steels showed the most definite directional properties. This might have been expected on the basis of the macrographic examination which was made of all steels by hot etching in hydrochloric acid. The structural silicon steels showed a particularly fibrous structure. It should be pointed out that the directional properties normal to the plane of the plate were not investigated.

SIGNIFICANCE OF THE TEST RESULTS

It is important in any survey of tensile and compressive characteristics of a material to consider what relation they may have to the problems of structural design.[18] Such was the purpose, in part, of the 1940 A.S.T.M. Symposium on the Significance of the Tension Test. The relation of unidirectional strength or ductility properties to the behavior of a structure is twofold:

1. The relation between the unidirectional properties and the properties under a state of combined stress.
2. The relation between the properties of a material, under a state of combined stress, and the properties of a structure, in which the state of stress may vary from point to point, and over-all deformations may be determined partly by shape characteristics.

A great deal of work remains to be done along these lines. The structural significance of the ratio of yield point or yield strength to ultimate strength and the structural significance of the general shape of the stress-strain curve beyond the elastic range are problems which are not fully understood as yet.

Some of the steels had upper yield points below the specification minimum. Some of these might have passed specifications if the maximum allowable testing speed had been used. It would appear, therefore, that a specification minimum yield strength should be discounted slightly in determining allowable working stresses.

The "Bauschinger" effect might be important structurally if a material which had been stressed beyond the yield point in tension were to be used in a compression member. The reduced effective modulus[19] of such a material would materially lower the critical buckling load in such a structural member.

The ductility requirements of a structural material have been a subject of much discussion but there is little definite knowledge available.[20]

The "true" indices of ductility proposed by MacGregor[3] represent a distinct step forward in quantitatively differentiating "uniform" and "necking" ductility. A profitable field of research is here opened to determine the relative structural significance of these two types of ductility.

The use of $\frac{3}{4}$-in. plate as the principal source of material for this investigation eliminates certain variables which might otherwise have affected the test results. The same material rolled thinner would have had different stress-strain characteristics. The same material rolled into a structural section such as an I-beam would have had nonuniform thickness resulting in initial strains due to cooling. In either case the alteration in stress-strain characteristics would have been due to differences in the previous strain history of the material.

CONCLUSIONS

The following conclusions apply only to the steels because there were insufficient samples of structural aluminum to form a basis for definite conclusions.

1. The average values of the upper and lower yield points of each type of steel tested in this program were practically the same in compression as in tension.
2. The general shape of the individual stress-strain curves up to the upper

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[18] Bruce Johnston, "The Structural Significance of Stress," *Civil Engineering*, Vol. 9, May, 1939, pp. 291–294.

[19] William R. Osgood, "Column Curves and Stress-Strain Diagrams," *Journal of Research*, Nat. Bureau Standards, Vol. 4, No. 4, October, 1932, pp. 571–582 (Research Paper No. 492).

[20] H. W. Gillett, "The Limited Significance of the Ductility Features of the Tension Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 551–579 (1940); also discussion, p. 601.

yield point and into the lower yield point range were very nearly alike in tension and compression, except that the proportional limits were usually higher in compression. The greatest differences between tension and compression stress-strain characteristics were noted across the direction of rolling in the silicon steels.

3. The upper yield point averaged 8.0 per cent higher in tension and 6.4 per cent higher in compression than the lower yield point with approximately the same difference for each class of steel.

4. The average ratio in tension between upper yield point and nominal maximum stress was 0.56 for carbon structural, 0.55 for silicon structural, and 0.65 for the low-alloy steels, with very little difference in the ratio with or across the direction of rolling in each case.

5. The direction of rolling affected the proportional limit, true breaking stress, and most of the ductility indices more than the other properties. Except for the proportional limit, the properties most affected were associated with the tensile behavior beyond maximum load.

6. The differences in directional properties were most marked in the case of silicon structural steel.

7. The rating of the three general types of steels with respect to various ductility criteria was as follows:

Elongation in 2 in., and "true" uniform index
  (1) Carbon structural steel.
  (2) Low alloy steel.
  (3) Silicon structural steel.
Reduction of area, and "true" necking index
  (1) Low alloy steel.
  (2) Carbon structural steel.
  (3) Silicon structural steel.
"True" over-all index of ductility
  (1) Low alloy steel.
  (2) Carbon structural steel.
  (3) Silicon structural steel.

8. The elastic constants were not affected by the direction of rolling and the average values for all tests were:

Modulus of elasticity, $E = 29,540,000$ psi.
Poisson's ratio, $\nu = 0.297$
Shearing modulus (calculated from E and ν)
$G = 11,390,000$ psi.

The average values of E_c and E_T were very nearly equivalent for every class of metal tested.

Acknowledgment:

The tests reported in this paper were part of a regular research project of the Fritz Engineering Laboratory, of which Hale Sutherland is the Director. Special acknowledgment is due to Howard J. Godfrey, Engineer of Tests, for his cooperation in the program. Equipment for the lateral electric strain gage was loaned by the Massachusetts Institute of Technology, through the cooperation of A. V. deForest. Funds contributed by the Chicago Bridge and Iron Co. for research in cooperation with the A.S.C.E. Committee on Design of Structural Members were used to build special test equipment. Materials were contributed by the Aluminum Corporation of America, the Bethlehem Steel Co., the Carnegie-Illinois Steel Co., the Otis Steel Co., and the Youngstown Sheet and Tube Co.

DISCUSSION

Mr. E. A. Davis[1] (*presented in written form*).—Messrs. Johnston and Opila have very carefully run their tests and, in general, have quite clearly described the results. There is one point, however, which may merit a little closer study. In describing the cyclic loading test shown in Fig. 5, they say that at (7) the initial tensile characteristics of the material have been restored. This may easily be misunderstood to mean that the test specimen is now back in the same condition as it was at the beginning of the test. The experience of the writer would indicate that the cyclic diagram (Fig. 5) can be explained in the following manner.

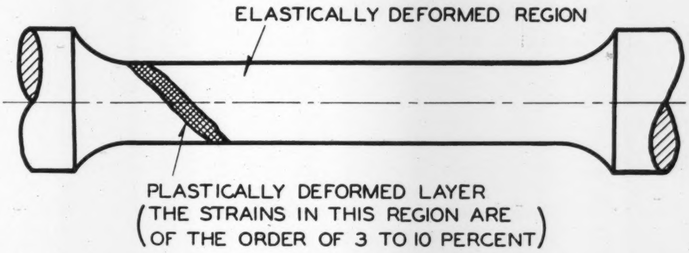
ELASTICALLY DEFORMED REGION
PLASTICALLY DEFORMED LAYER
(THE STRAINS IN THIS REGION ARE OF THE ORDER OF 3 TO 10 PERCENT)

Fig. 1.—Deformation in a Mild Steel Bar Just After the Lower Yield Point Is Reached.

It can be seen that the bar was unloaded (2) very soon after the first localized slip occurred. This slip layer would probably be located as shown in the accompanying Fig. 1 of this discussion. This means that a large part of the specimen was still in a purely elastic state. If it were reloaded in tension after the unloading (2) a loop similar to that at (6) and (7) would be obtained and when a stress somewhat higher than the lower yield point were reached, localized slip would be resumed. This would cause the stress to drop to the lower yield point and would transform more of the elastic portion into the plastic state. The "Bauschinger Effect" noticed in the compression loading (3) would necessarily have to be contributed by that part of the material already in the plastic state, for material which has not been deformed plastically does not show this effect. The drop in load at the lower end of the compression loading (3) indicates that localized yielding is taking place in some of the remaining elastic regions. This same description will hold for the steps five, six, and seven.

The total yield point elongation, or that elongation occurring in what the authors call the lower yield point range depends upon such factors as composition and speed of deformation and may be as high as 10 or 12 per cent. If we assume that for this material it is as low as 3 per cent, the flat portion of the curve in the lower yield point region should spread over about thirty-six of the units of strain shown in Fig. 5. It seems that several more such cycles might have been made before the localized yielding or "drop of the beam" phenomena would completely disappear, yet the material is definitely not in its original condition.

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[1] Research Engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

MR. MARSHALL HOLT[2] (*presented in written form*).—The authors have undertaken an important study of structural materials and have made a valuable contribution to the store of test data on the behavior of commonly used structural alloys. There are, however, some points in the paper that seem to need further discussion.

The cyclic stress-strain curves for 27S-T in Fig. 6 are misleading because of the use of the dash line, a type of line used to indicate an extrapolation in the case of some of the stress-strain curves for steel, for example see Fig. 2. It appears that the dash line in this case merely shows an offset in the curves to avoid confusion. The cyclic stress-strain curves for 27S-T are known to be very similar to those for aluminum alloy 17S-T shown in Fig. 13 of "Some Stress-Strain Studies of Metals," by R. L. Templin and R. G. Sturm.[3]

From the few cyclic stress-strain tests made it is concluded that the "Bauschinger effect" would be detrimental, that is, the loading beyond the yield strength in one direction would always result in a decrease in the yield strength in a subsequent loading in the opposite direction. It is shown by Templin and Sturm that with larger amounts of stretching both the tensile and compressive yield strengths are increased in the case of the aluminum alloys.

The use of Huggenberger tensometers with such a short gage length is usually not considered good practice for determining the modulus of elasticity; however, the reported values of modulus of elasticity of 27S-T are in good agreement with the results of tests made on the same lot of material by the Aluminum Research Laboratories using a longer gage length and an extensometer of greater precision. The good agreement of the tensile properties of 27S-T determined by the Aluminum Research Laboratories with those reported in Table II of the paper is shown below:

	Aluminum Research Laboratory		Lehigh University	
	With Grain	Across Grain	With Grain	Across Grain
1-in. plate:				
Tensile strength, psi.	60 300	59 800	59 500	58 700
Yield strength, psi.	40 700	40 600	40 500	40 300
Elongation in 2 in., per cent	15.1	13.3	17.5	13.5
1-in. rod:				
Tensile strength, psi.	66 500		68 300	
Yield strength, psi.	56 280		53 500	
Elongation in 2 in., per cent	12.8		16.5	

The assumption is made by the authors that the use of $\frac{3}{4}$-in. thick plate eliminates enough of the variables affecting the properties that material with uniform properties would be obtained. The extent to which uniformity is attained can be determined from a study of the following ranges in tensile properties of carbon steel and silicon steel found in Table II:

Property	Range of Values for Carbon Steel	Range of Values for Silicon Steel
Tensile yield strength, psi.		
With grain	31 200 to 41 900	41 500 to 49 200
Across grain	30 300 to 38 700	42 400 to 48 700
Tensile strength, psi.		
With grain	56 000 to 64 700	80 600 to 94 200
Across grain	55 000 to 72 500	81 400 to 90 100
Elongation in 2 in., per cent		
With grain	37.0 to 45.5	28.5 to 36.5
Across grain	28.0 to 42.0	16.5[a] to 32.5

[a] Broke near punch mark.

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[2] Research Engineer, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[3] *Journal of Aeronautical Sciences*, Vol. 7, No. 5, March, 1940, pp. 189–198.

These variations would undoubtedly be satisfactory for materials for usual commercial use, but do the authors consider them satisfactory for material for the research program undertaken?

MR. A. NADAI[4] (*by letter*).—With regard to the test results shown in Fig. 4 for the upper yield point obtained in compression tests in which the cylinders had a ratio of the length L to the diameter D, which varied between the values $L/D = 1$ to 6, the authors of the paper point to the end effects at the compression plates. The writer recalls that in some compression tests made several years ago with mild steel, conical surfaces of shear could be demonstrated in the interior of the cylinders by means of the Fry etching method. First yielding in the steel cylinders occurred along conical surfaces which started at the edges of the test cylinders. It is believed that yielding along such surfaces is responsible for the first drop of the load when well-centered and ground steel cylinders are compressed in the axial direction between perfectly smooth parallel plates and that the "first yielding" in a cylindrical test specimen in tension and in compression may be due to different not homogeneous states of stress, since it is practically impossible to produce an undisturbed state of axial compression due to the friction of the compression plates.

MR. J. M. LESSELLS[5] (*by letter*).—The paper is of considerable interest because it brings together test data on structural carbon and silicon steels and low-alloy steels.

Apart from the statistical data presented, the authors discuss certain features such as upper and lower yield point, Bauschinger effect, overstrain and true stress-strain relations but these do not appear to add anything to the known knowledge.

On page 566, there is a statement that "the lower yield point is considered by some to be a more stable criterion." This could be more positive since most engineers believe that the lower yield point is the more important physical property.

As regards overstrain effects, Fig. 7 shows recovery effected by boiling in water. It would have been interesting to have had similar data on the alloy steels. Mention should also be made of Dalby's[6] work.

Directional properties are of importance. There are indications that certain heats of alloy steel show a distinct drop in transverse properties which can only be shown by fatigue tests.

MR. H. J. GODFREY[7] (*by letter*).—This significant paper on the physical properties of structural alloys emphasizes the importance of using the utmost accuracy in carrying out the simple tension and compression test. The data presented should be of great assistance in determining the meaning of the various physical measurements which are associated with the physical characteristics of structural alloys.

The use of the "true" ductility indices to evaluate the physical properties of structural alloys is of extreme interest, particularly in regard to the effect of the direction of rolling on the necking ductility index.

The writer has had the opportunity to make a limited number of bend tests on some of the material included in this paper and these tests were also affected by the direction of rolling. The bend-testing machine used for these tests was designed initially for the testing of steel wire. A description of the machine and its operation may be found in a paper by the writer on "The Fatigue and Bending Properties of Cold-Drawn Steel Wire."[8] In brief, the test consists of bending the test specimen back and forth over rollers until fracture occurs. During the bend test a small tension load is applied to the specimen.

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[4] Consulting Mechanical Engineer, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[5] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[6] "Strength and Structure of Steel and Other Metals," Longmans, Green, and Co.

[7] Development Engineer, Development Engineering Laboratory, John A. Roebling's Sons Co., Trenton, N. J.

TABLE I. BENDING PROPERTIES OF STRUCTURAL ALLOYS.

Material	Test Number[a]	Necking Indices	Number of 180-deg. Bends	
			Specimen No. 1	Specimen No. 2
Carbon structural	11A	0.868	38.0	36.0
	11B	0.598	27.5	23.5
Silicon structural	16A	0.703	38.0	38.5
	16B	0.447	21.5	18.5
	17A	0.603	30.0	31.5
	17B	0.598	25.5	26.0
	18A	0.828	43.5	38.0
	18B	0.471	18.5	19.5
Low alloy A-3	24A	1.122	40.5	41.0
	24B	0.758	25.5	27.0
Aluminum 27S-T	34A	0.037	4.5	3.5
	34B	0.029	3.5	2.5
	35A	0.310	10.5	9.5

[a] A = specimens tested with the direction of rolling;
B = specimens tested across the direction of rolling.

The bend specimens were machined from $\frac{3}{4}$-in. plates to a cross-section of 0.20 by 0.10 in. and were approximately 6 in. in length. The specimens were bent through 180 deg. in their weakest direction over rolls 1 in. in diameter with the specimen so placed that the roll diameter was ten times the thickness of the specimen. The results of the bend test are presented in the accompanying Table I along with the corresponding necking indices reported in the paper. The direction of rolling was found to have considerable effect on the number of bends to failure and in every case the specimens tested with the direction of rolling withstood a greater number of bends than the corresponding specimens tested across the direction of rolling.

These test results are also presented in the accompanying Fig. 2 where a direct relationship between the necking ductility index and the number of bends may be observed. The structural aluminum alloys which had a relatively low necking ductility index also withstood very few 180-deg. bends as compared to the structural steel specimens.

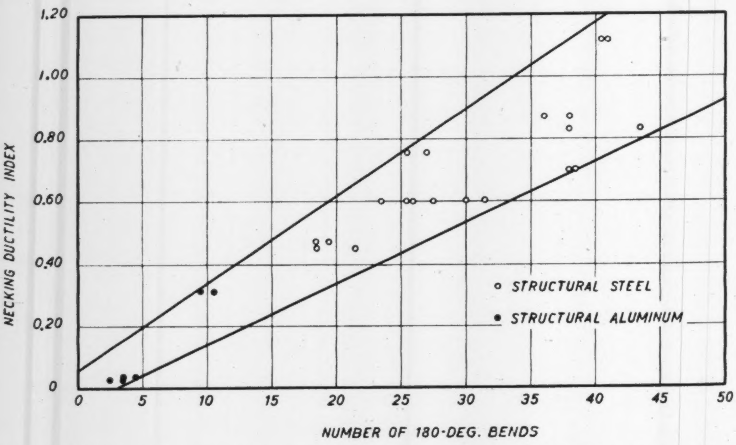

Fig. 2.—Relation Between the Necking Ductility Index and the Number of Bends.

MR. W. LEIGHTON COLLINS[9] (*by letter*).—It is quite evident that one of the purposes of the work of Messrs. Johnston and Opila was to compare the action of some of the newer low-alloy high-strength steels in tension and compression to determine whether their behavior is the same as that of a number of more common and more widely used plain carbon steels under the same types of loading. The tests were conducted with unusually great care in regard to axiality of loading, and the comparisons made indicate that there are no great differences in the tensile and compressive properties of the steels tested and that the behavior of the two general types of steels, when stressed in tension and compression, are essentially the same. Only unidirectional stress conditions were considered by the authors. Other states of stress, such as in a torsion specimen or a notched tensile specimen, may be more significant in ascertaining differences in the behavior of materials subjected to loads.

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[8] *Transactions*, Am. Soc. Metals., Vol. XXIX, No. 1, March, 1941, p. 133.

[9] Assistant Professor, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

TABLE II.—MECHANICAL PROPERTIES OF LOW-ALLOY HIGH-STRENGTH STEELS.

Each value is the average of 3 tests, except as noted.

All specimens came from ¾-in. round bars in the "as-rolled" condition

Material	σ_P, psi.[a]	σ_{UP}, psi.		σ_{NM}, psi.		Modulus of Elasticity, psi.	Elongation, per cent	Reduction of Area, per cent	Ratio, $\frac{\sigma_P}{\sigma_{UY}}$	Ratio, $\frac{\sigma_{UY}}{\sigma_{NM}}$	Ratio, $\frac{\sigma_{UY}\text{ Com.}}{\sigma_{UY}\text{ Tens.}}$[b]
Tension—D = 0.500 in. 2-in. gage length. Shoulder-type specimen.											
A	50 000	58 000		76 900		29 800 000	40.2	73.1	0.86	0.76	....
B	48 400	55 800		71 200		29 600 000	38.0	69.8	0.87	0.78	....
C	48 300	62 800		92 200		29 500 000	29.5	54.8	0.77	0.68	....
D	62 500	83 700		128 000		31 000 000	23.1	58.3	0.75	0.65	....
E	31 300	51 300		77 100		28 800 000	41.3	72.3	0.61	0.66	....
36	32 800	35 500		59 300		30 300 000	41.6	68.6	0.92	0.60	....
Compression—D = 0.750 in. 2-in. gage length. Fixed heads with parallel machined ends.											
		$\frac{L}{D} = 3$	$\frac{L}{D} = 6$	$\frac{L}{D} = 3$	$\frac{L}{D} = 6$						
A	60 600	65 300	65 100	199 000[c]	75 000	32 000 000	....	....	0.93	....	1.13
B	54 700	59 300	59 400	169 000[c]	70 600	30 300 000	....	....	0.92	....	1.06
C	52 900	64 900	65 200	181 000[c]	87 800	29 400 000	....	....	0.81	....	1.03
D	71 000	85 900	85 400	227 000[c]	121 000	31 400 000	....	....	0.83	....	1.02
E	29 700	49 300	49 500	185 000[c, d]	69 500[d]	28 000 000	....	....	0.60	....	0.96
36	37 800	37 800	38 500	....	58 000	30 600 000	....	....	0.98	....	1.06

[a] Stress at departure from initial straight line.

[b] For $\frac{L}{D} = 3$.

[c] Stress limited by range of movement of compression rig.

[d] One test only.

The writer has made a number of tests on five low-alloy high-strength steels and one plain carbon steel to compare their actions in tension and compression. The tests were made without exercising as great care as the authors to insure axial loads, but nevertheless the tests were carefully made. The data were briefly referred to in the paper on "Physical Properties of Four Low-Alloy High-Strength Steels"[10] and are presented in their entirety in the accompanying Table II. It is felt that a comparison of the data from the very carefully conducted tests for 1-in. gage lengths with the data obtained from what may be a more common method of testing without undue precautions regarding axiality of loading would be of some value.

For all the tests reported in Table II an averaging extensometer having a gage length of 2 in. and a multiplication ratio of 5:1 (1 division on the Ames dial equals a strain of 0.0001 in. per inch) was used. Materials A, B, C, D, and E are low-alloy high-strength steels and 36 is a plain carbon structural steel, all but E being the same steels as reported on in the paper already referred to.[10] The tension test specimens conformed with A.S.T.M. Tentative Methods of Tension Testing of Metallic Materials (E 8 – 40 T)[11]; the compression specimens were 0.75 in. in diameter, only the ends being machined. The rig used for making the compression tests was similar to that shown in Fig. 3 of A.S.T.M. Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T).[12]

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[10] W. L. Collins and T. J. Dolan, "Physical Properties of Four Low-Alloy High-Strength Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 157 (1938).

In interpreting data for modulus of elasticity of steel it must be borne in mind that the use of a gage length of less than 8 in. is usually undesirable. However, in order to compare tensile and compressive properties for specimens of equal diameters a gage length of less than 8 in. must be used, particularly if the specimens are to be kept small. The fact that some confidence can be had in the comparisons based on carefully made tests using gage lengths of less than 8 in. is borne out by the data for materials A, B, C, D, and 36 included in Table II and in the paper already referred to.[10] The moduli of elasticity determined for gage lengths of 2 in. varied from −5 to +3 per cent, average of +1 per cent, from the values obtained for gage lengths of 8 in.

For the six steels studied by the writer, very little difference in the general shapes of the stress-strain curves in tension and compression for any given material could be observed for stresses up to the yield points. However, the proportional limits, yield points, and moduli of elasticity usually have higher values in compression than in tension, the average differences being 10.6, 4.7, and 1.5 per cent, respectively. The authors report no differences in yield point and modulus of elasticity.

In discussing the proportional limit of a material it must be remembered that it is a purely arbitrary value for a given machine, extensometer, scale of plotting, etc.[13], and should not be considered to be a definite property of a material. The values for proportional limit are given in the accompanying Table II only to afford a comparison with the authors' results; little confidence should be placed in them as a property of a material or as measuring any particularly significant action of the materials.

The so-called proportional limit of the authors is really a stress corresponding to a very small offset, "less than about 0.00001 in." as the authors say. The stress at a small offset, or several offsets, is of value, along with yield point or yield strength, in providing information regarding the shape of the stress-strain curve in the region of departure from a straight line, but since the 0.00001 in. used is also the limit of the sensitivity claimed for the Huggenberger tensometers, doubt must be attached to the significance of stresses so determined and to the ability of others to reproduce them. It is desirable to omit the proportional limit as a property of the material and in its place give, perhaps, the stress at a small definitely measurable offset which is considerably larger than the smallest reading of the extensometer. The latter method would give a stress that most probably could be reproduced by others, that would be a measure of some significant action of the material and that would not be influenced greatly by the many variables affecting the proportional limit as it is ordinarily defined and as stated in the A.S.T.M. Standard Definitions of Terms Relating to Methods of Testing (E 6 - 36).[14]

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[11] 1940 Supplement to Book of A.S.T.M. Standards Part I, p. 453.

[12] 1939 Book of A.S.T.M. Standards. Part I, p. 1230.

[13] R. L. Templin, "The Determination and Significance of the Proportional Limit in the Testing of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 523 (1929).

MESSRS. BRUCE JOHNSTON[15] AND FRANCIS OPILA[16] (*authors' closure*).—The discussion by Mr. Davis provides a very clear explanation of the action during alternate cycles of compression and tension for a material such as structural steel which has a flat stress-strain diagram in the lower yield-point range. The authors state that in the cyclic loading test shown in Fig. 5, at (7) "restoration of initial tensile characteristics" occurred. As Mr. Davis points out, this might be misinterpreted to imply that the material was restored to its original condition, which of course is not the case. The authors agree entirely with all of Mr. Davis' comments.

Mr. Holt is correct in his remarks about the dash line used to offset the curves in Figs. 5 and 6 to avoid confusion. In the case of Fig. 5 the dashed line also indicates that the exact amount of strain in the lower yield point range could not be measured. In Fig. 6, on the other hand, the dashed line does not represent strain at all, but is simply an arbitrary offset used to separate the curves. The authors appreciate the corroboration of their test results on structural aluminum by tests as made by Mr. Holt. In the last two paragraphs of his discussion, Mr. Holt apparently misunderstands one of the main purposes of the test program. Uniformity of material was consciously avoided and the steels were obtained from four different manufacturers. Each of the 33 samples represented a different heat of steel. A commercial sampling of a variety of steels was thereby obtained and the results show to what extent variation may be expected. The results also show the agreement between tensile and compressive properties both with and across rolling for any particular sample of steel. When steel is used for research in which the material itself is not a variable it is obviously desirable to obtain material from one heat and one rolling so as to get more uniform physical properties.

Mr. Nadai contributes some interesting observations about probable differences between the stress raisers in tension and compression which contribute to initial yielding.

Mr. Lessells states that "most engineers believe that the lower yield point is the more important physical property." The authors are glad if this is true, because they agree in this opinion. Nevertheless, the standard acceptance tests of structural steel make use of the upper yield point. Even in research papers the lower yield point is often omitted in the tabulation of physical properties. At least among the structural engineers the upper yield point is frequently the accepted criterion of yield strength.

Mr. Lessells points out that a distinct drop in the transverse properties of alloy steels may sometimes be shown by fatigue tests. Mr. Godfrey, in his discussion, provides valuable additional information on this question by reporting repeated bend tests on some of the same samples of material as were used by the authors. These repeated bend tests may be considered as a type of highly accelerated fatigue test in which the stress magnitudes are far above the yield strength of the material. Mr. Godfrey's bend test is shown to bring out important properties of metal which are not indicated by the usual indices of strength and ductility as determined by the standard tension test. The correlation in Fig. 2 of the "true" index of necking ductility with the results of the repeated bend test is particularly interesting and indicates a profitable field for future research.

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[14] 1939 Book of A.S.T.M. Standards, Part I, p. 778; Part II, p. 871; Part III, p. 553.

[15] Associate Director, Fritz Engineering Laboratory, and Associate Professor of Civil Engineering, Lehigh University, Bethlehem, Pa.

[16] Formerly Lawrence Calvin Brink Research Fellow in Civil Engineering, Lehigh University; now associated with Baldwin-Southwark Corp., Chicago, Ill.

Mr. Collins provides information similar to that obtained by the authors on a number of low-alloy steel samples.

The authors appreciate the interest which has been shown in this paper by the various discussers. The discussions have added materially to the information on the subject of tension and compression tests. Although careful tests in tension and compression have been made again and again during the past seventy-five years, the continual changes in types of materials make a repetition of such work desirable from time to time. In addition, the introduction of more adequate ductility criteria in recent years has extended the scope of the information to be obtained from the simple tension test.

After this paper went to press it was brought to the authors' attention that it would be desirable to differentiate between universal mill plate, which is usually rolled in one direction only, and sheared plate, which undergoes varying degrees of transverse rolling in the preliminary passes. The mills have furnished information in this respect which has enabled the following classification as to type of rolling of all samples excepting the 1 in. thick specimens.

1. Universal mill, or hot strip mill plate, in general, rolled in one direction only. Tests Nos. 4, 8,[17] 12, 13, 14, 15, 16, 23, 30, 32[18] and 33.[18]

2. Sheared plate, which received preliminary passes in a direction transverse to the final rolling direction. The relative amount of cross-rolling varies with the final plate dimensions desired. Tests Nos. 1, 2, 3, 5, 6, 7, 9, 10, 17, 19, 20, 21, 22, 25, 26, 27, 28, and 29.

There are not enough tests available to justify the tabulation of averages. The carbon steel samples were predominantly sheared plate whereas only one sample of the silicon steel was sheared plate.

The two carbon structural steel samples in type 1 showed the greatest directional effect of any of the carbon steels, both as to yield point and ductility criteria. The one sample of sheared (type 2) silicon steel showed the least directional effect of the silicon steels.

The low alloy steels, of which nine are known to be type 2 and four type 1, gave contradictory evidence. Three of these type 1 steels (30, 32, and 33) show no directional effect whatever with respect to yield point, contrary to what might be inferred from the behavior of the structural and silicon steels. The fourth type 1 steel, in this general group, showed the greatest directional effect, as would be expected.

It may be concluded that steels which have a tendency toward developing certain directional effects are likely to have this tendency accentuated when rolled in a universal mill, or in one direction only. This conclusion applies principally to yield point and ductility criteria. The nominal ultimate tensile strengths were not appreciably affected by direction of rolling, type of rolling, or type of steel.

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[17] Angle specimen, rolled in one direction only.

[18] Hot strip mill plate.

THE FABRICATION OF CARBON-MOLYBDENUM PIPING FOR HIGH-TEMPERATURE SERVICE

By R. W. Emerson[1]

Synopsis

The present method of acceptance of carbon-molybdenum piping for high-temperature service is based on metallographic examination for grain size and structure.

Though there appears to be general agreement as to the proper grain size for increased creep strength, there also appears to be disagreement as to the rate of cooling of pipe from the heat-treating temperature.

This paper deals with the effects of hot fabrication such as upsetting, hot bending, forging, and welding on the physical and metallographic properties of low carbon - 0.5 per cent molybdenum steel (A.S.T.M. Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 40 T)[2]), and the removal of same by final heat treatment for grain size. The effect of heat-treating temperature, cooling rate, and steel melting practice on grain size and structure of this material is also discussed.

Since welding follows final heat treatment for grain size and since a variety of microstructures exists in and adjacent to the welded joint, many of which would be considered as unsatisfactory if found in the pipe proper, the question is asked: "If the creep properties in or adjacent to the welded joint are somewhat inferior to that of the piping proper, or at least the microstructure considered as such, of what significance is this problem?"

Today modern power plants are being designed for operating temperatures as high as 950 F. and working pressures as high as 2000 psi. Essentially continuous operating conditions such as these, with the design stresses used, will produce over a period of time, plastic flow in low carbon - 0.5 per cent molybdenum steel. Considerable pioneering has been done on the subject of creep (plastic flow of metals at elevated temperatures) and experience has shown that the variables which may possibly affect the creep rate of steel are not only multitudinous, but illusive in nature. Of the many variables which appear to affect the creep rate of both carbon and alloy steel, and of which considerable research has been done, ferrite grain size[3] and carbide structure have appeared to be outstanding (**1, 2, 3, 4**).[4]

Final acceptance of carbon-molybdenum piping for high-temperature service is based on metallographic examination for grain size and carbide structure. There seems, at the present time, to be general agreement to the fact that for increased creep strength, a medium grain size (A.S.T.M. Nos. 3 to 6) is to be preferred to a fine grain size (A.S.T.M. Nos. 7 to 8). There also appears, at this time, however, to be some disagreement as to the form of the carbide structure for best creep results, since in some cases furnace-cooled pipe is specified, while in others, air cooling is specified. In the former instance, a dense sorbitic pearlite is formed; the latter treatment, however, producing a Widmanstaetten carbide structure.

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[1] Metallurgist, Pittsburgh Piping and Equipment Co., Pittsburgh, Pa.

[2] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 252.

[3] The term "grain size" when used hereafter refers to ferrite and carbide grain size and not austenite grain size, unless so stated.

[4] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 604.

It is not the object of this paper to take any definite stand as to the proper heat treatment of low carbon - 0.5 per cent molybdenum steel piping (A.S.T.M. A 206) in order to obtain maximum creep strength, but rather to discuss the effect of the hot-fabricating processes, including final heat treatment, on the physical and metallographic properties of this material. It is the further object of this paper to leave with the reader certain questions which at the present time appear puzzling to the piping fabricator.

The detailed subjects to be considered are the following:

1. The metallurgical response of low carbon - 0.5 per cent molybdenum steel piping to heat treatment.
2. The microstructure and physical properties of carbon-molybdenum steel piping as affected by:
   (*a*) Hot bending.
   (*b*) Upsetting.
3. The effect of finishing temperature and percentage of hot reduction on the grain size and carbide structure of forged carbon-molybdenum steel.
4. The microstructure and physical properties of welded joints.
5. The effect of the heat-treating temperature, cooling rate, and steel melting practice on final grain size and carbide structure.

STEELS INVESTIGATED

The analysis and previous fabrication of the steel used in this investigation are given in Table I. Though all material used, met the chemical requirements of Specifications A 206 – 40 T, the grain size requirements of the above specifications could be obtained only with heats Nos. 1 to 5, No. 6 having an A.S.T.M. grain size of Nos. 6 to 8 after annealing at 1750 F.

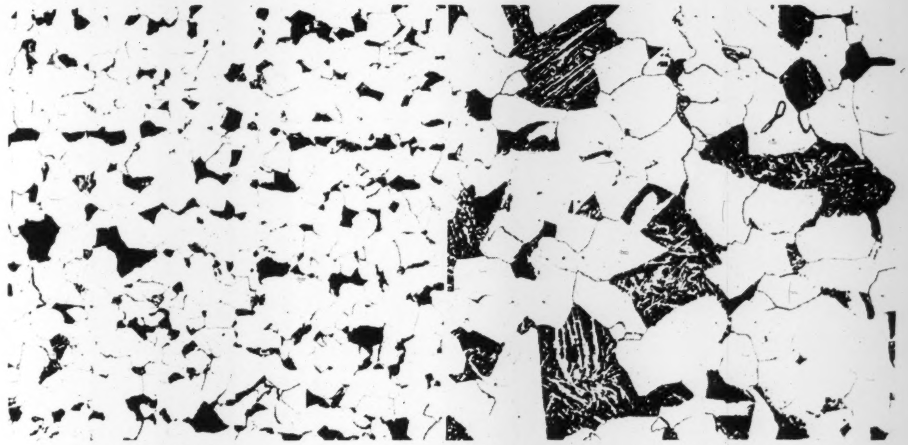
TABLE I.—CHEMICAL ANALYSES AND PREVIOUS FABRICATION OF STEEL INVESTIGATED.

Heat	Composition, per cent						Previous Fabrication
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Molybdenum	
No. 1	0.17	0.62	0.010	0.021	0.25	0.50	From 12-in., 160 schedule pipe
No. 2	0.17	0.53	0.015	0.015	0.24	0.52	From 4-in., 120 schedule pipe
No. 3	0.18	0.53	0.013	0.018	0.18	0.56	From 10-in., 120 schedule pipe
No. 4	0.18	0.46	0.020	0.022	0.16	0.50	From 22½ by 13½ by 28½-in. forging
No. 5	0.17	0.60	0.015	0.015	0.20	0.53	From 8 by 8-in. billet
No. 6	No analysis						From 10-in., 100 schedule pipe

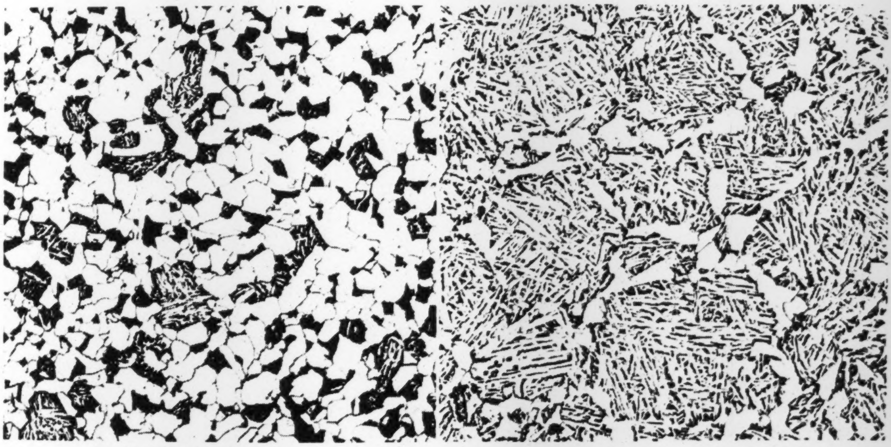
The specimens used for experimental purposes were taken from hot-rolled pipe, a large forging block, an 8 by 8-in. billet section, and a 3 by 3-in. section forged from a portion of the 8 by 8-in. billet.

THE METALLURGICAL RESPONSE OF LOW CARBON - 0.5 PER CENT MOLYBDENUM STEEL PIPING TO HEAT TREATMENT

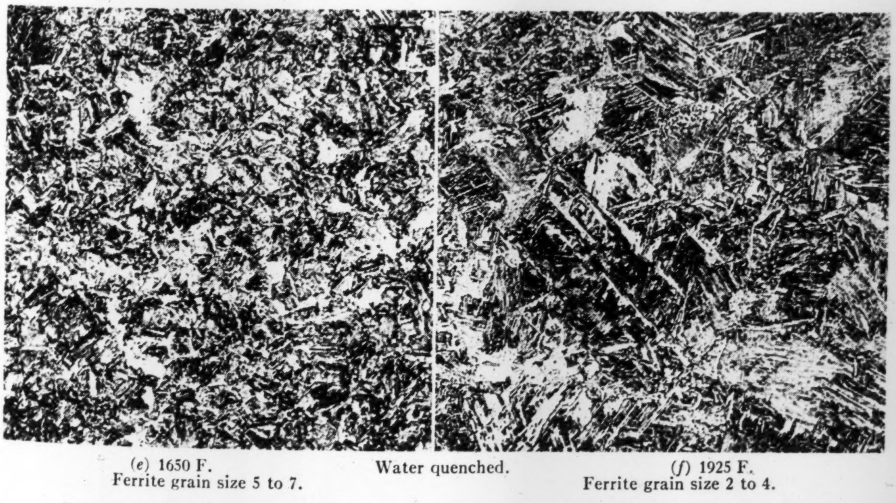
The heat treatments involved in the hot fabrication of carbon-molybdenum piping may vary from an annealing treatment to one which approaches a water



(a) 1650 F. Furnace cooled. (b) 1925 F.



(c) 1650 F. Air cooled. (d) 1925 F.



(e) 1650 F. Water quenched. (f) 1925 F.
Ferrite grain size 5 to 7. Ferrite grain size 2 to 4.

FIG. 1.—Microstructure of Low Carbon - ½ per cent Molybdenum Steel (× 100).

quench. In view of the above possible variations in heat treatment, it is essential that the response of this material to heat treatment be known.

Photomicrographs of samples held at 1650 F. and 1925 F. for 2 hr. per inch of thickness followed by (*a*) furnace cooling, (*b*) air cooling, and (*c*) water quenching are shown in Fig. 1. The size of the specimens used for heat treatment were 1 by 1.3 by 2 in. and were removed from the 12-in., 160 schedule pipe, heat No. 1, Table I. It was found that the test results from the use of specimens of this size more closely approximated parallel conditions in actual fabrication than did smaller specimens. These specimens were prepared to check both hardness and microstructure with that found in pipe which had undergone actual fabricating conditions. The average converted Brinell hardness of each of the six specimens is shown in Table II.

TABLE II.—AVERAGE CONVERTED BRINELL HARDNESS OF LOW CARBON – 0.5 PER CENT MOLYBDENUM STEEL.

Heat-Treating Temperature, deg. Fahr.	Furnace Cooled	Air Cooled	Water Quenched
1650	126	140	302
1925	126	166	293

It is to be observed that variation in heat-treating temperature had essentially no effect on the hardness of the furnace-cooled specimens and only a minor effect on the microstructure. The higher heat-treating temperature resulted in increased grain size and a decreased density of the sorbitic pearlite. For relatively coarse-grained material such as that of Fig. 1(*b*), a slower rate of cooling is necessary than for that of Fig. 1(*a*), to produce pearlite of equal density to that of Fig. 1(*a*).

Fig. 1(*c*) and (*d*) are representative of the Widmanstaetten carbide structure obtained by the air cooling of this material. With this heat-treatment, the higher temperature resulted in a structure in which the dense sorbitic pearlite was completely absent (complete Widmanstaetten structure). However, as in the case of the annealed samples, the specimen having the more coarse grained structure, had the pearlite of least density. Variation in the normalizing (air-cooling) temperature was noted to produce a slight variation in the hardness as might be expected from the variation in the microstructure. The hardness in either case, however, is considered to be within a range which would indicate satisfactory ductility.

The microstructure produced from water quenching carbon-molybdenum steel as may be observed from Fig. 1(*e*) and (*f*) is acicular and consists of ferrite and martensite. This material, in the quenched condition, may vary in Brinell hardness from 250 to 400 depending on the size of the specimen, quenching medium, etc., upon which the hardness of many quenched steels depend. Under drastic quenching conditions, the microstructure will be found to be completely martensitic. Since the carbon content of the material under discussion does not exceed 0.20 per cent, even in view of the 0.50 per cent of molybdenum present, the subcritical transformation rate is relatively rapid. It is doubtful whether the average hardness of a quenched specimen would approach the upper limits of hardness given with dimensions in excess of 0.75 in. per side.

The Microstructure and Physical Properties of Carbon-Molybdenum Steel Piping as Affected by Hot Bending and Upsetting

Hot Bending:

In the production of carbon-molybdenum pipe bends, straight lengths of pipe are filled and packed with sand. These are in turn heated to 1800 to 2000 F. bending temperature and placed on the bending table. Bending first occurs in the pipe adjacent to the bending shoes. When the proper radius has been obtained in this portion of the heated pipe, it becomes necessary to stiffen the pipe at this point in order to force bending to occur successively along

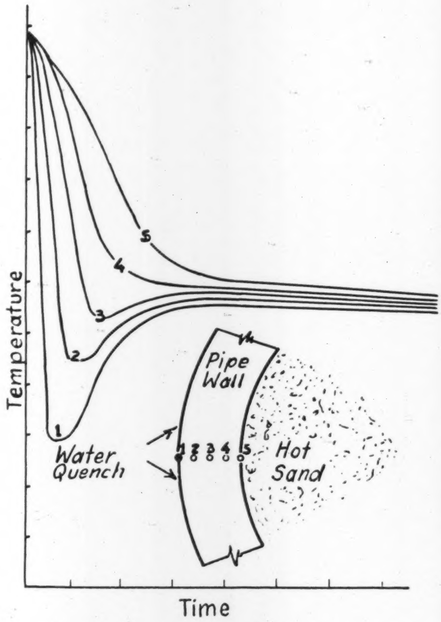


FIG. 2.—Schematic Cooling Conditions of Pipe Wall During the Bending Process.

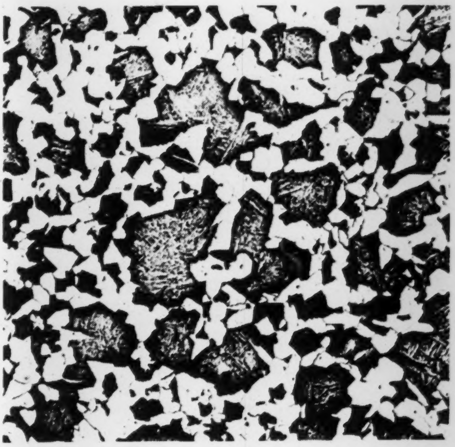
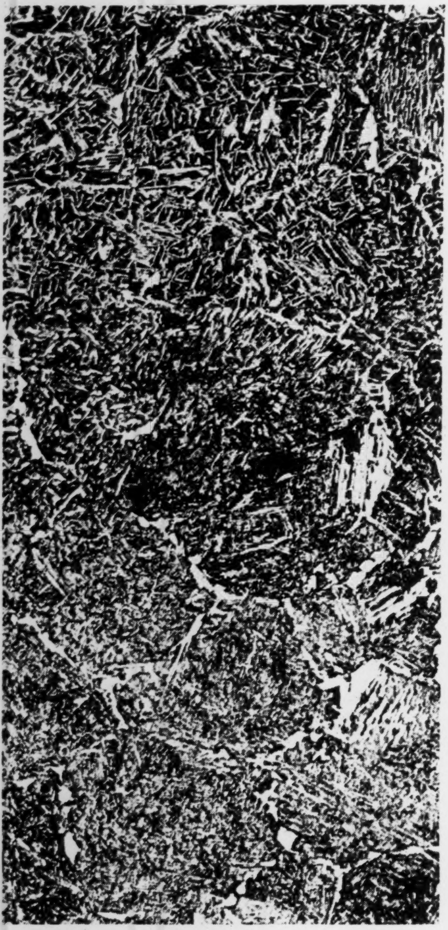


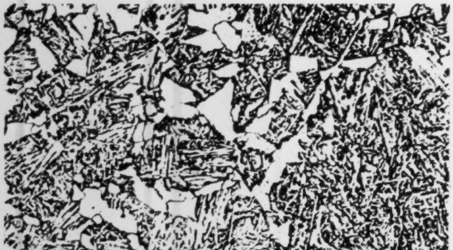
FIG. 3.—4-in., 120 Schedule Pipe, As-Bent Condition (× 100).

TABLE III.—PHYSICAL PROPERTIES OF HOT-BENT AND UPSET PIPE BEFORE AND AFTER HEAT TREATMENT.

Heat Treatment	Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Converted Brinell Hardness		
					Min.	Max.	Avg.
12-in., 160 Schedule Pipe							
As-bent condition	68 950 67 840	85 160 85 260	22.0 21.5	64.0 63.5	159	195	181
As-bent condition plus normalized at 1750 F.	45 380 56 940	76 050 78 760	26.0 25.0	62.6 64.4	153	172	165
As-bent condition plus annealed at 1750 F.	33 380 35 990	64 140 67 420	30.0 30.0	60.0 57.1	110	118	115
4-in., 120 Schedule Pipe							
As-bent condition	....	....	....	....	166	248	207
10-in., 120 Schedule Pipe							
As-upset condition	62 150 59 650	78 300 78 450	22.0 23.5	61.8 59.7	135	185	156
As-upset condition plus normalized at 1750 F.	65 300 56 720	80 090 82 630	22.0 25.0	64.7 65.9	148	170	161
As-upset condition plus annealed at 1750 F.	35 050 36 440	65 060 65 220	29.0 30.0	60.6 60.5	112	128	123



(*a*) Adjacent to outside diameter of pipe.



(*b*) Adjacent to inside diameter of pipe.

FIG. 4.—12-in., 160 Schedule Pipe, As-Bent Condition (× 100).

the heated portion of the pipe. Stiffening of the pipe is accomplished by water quenching using a 2-in. diameter stream of water. Quenching of the pipe to a black heat is sufficient to get requisite stiffening.

Since the pipe is quenched to only a black heat and the mass of sand on the inside is at a temperature considerably above that of the outside of the pipe the imposed cooling conditions are not so severe as it would first appear. The heat treatment imposed upon hot bent pipe is essentially a medium- to high-temperature sub-critical quench followed immediately by drawing at a temperature slightly above that to which the pipe was quenched. The cooling conditions at various points across the wall of a pipe during and after quenching is shown in Fig. 2.

The microstructure of a 4-in., 120 schedule pipe bend is shown in the as-bent condition in Fig. 3. This structure appeared essentially uniform from the outside to the inside diameter of the pipe at a magnification of 100 diameters. The average Brinell hardness across the wall of the pipe in the above condition, did vary however, from 166 to 248 (inside to outside diameter) as shown in Table III.

Observation at 1000 diameters magnification indicated this variation in hardness to be the result of an extremely small variation in transformation temperature of the carbon-rich austenite grains during quenching of the pipe bend.

Considerable variation in microstructure results however from the quenching of heavy-wall pipe as is shown in Fig. 4. The microstructure shown, was obtained from a specimen which was removed from a pseudo-bent, $12\frac{3}{4}$ in. in outside diameter by 1.312-in. wall pipe, the analysis of which is given in Table I. A 12-in. length of this pipe was packed with sand, heated to bending temperature and water quenched to a black heat and then allowed to self-anneal from the heat of the sand. After slow cooling to room temperature as a result of self-contained heat, the sand was removed. The pipe was of such a length that bending was not attempted.

From Fig. 4(*a*) it may be seen that the quenching action was sufficiently drastic on the outside of the pipe, in conjunction with a coarse-grain structure resulting from the high bending temperature, to cause essentially all ferrite to precipitate in the cleavage planes of the austenite rather than at the austenite grain boundaries. The water quenching of pipe with hot sand on the inside is in reality a gradient quenching operation. This being the case, the cooling rate at some point across the wall of the pipe should be such as to cause proeutectoid ferrite rejection at the austenite grain boundaries and thus reveal the austenite grain size. This was found to occur 0.18 in. from the outside of the pipe. The austenite grain size prior to the quenching operation, is shown in Fig. 4(*a*) to be A.S.T.M. No. 2. The structure of the pipe wall adjacent to the sand is shown in Fig. 4(*b*) and may be seen to be that of a relatively high temperature normalize. (Note similarity between Figs. 4(*b*) and 1(*d*).) Irrespective of microstructure variation, the hardness was found to vary only a small amount. The Brinell hardness varied uniformly from inside to outside diameter from 159 to 195. The as-bent pipe after normalizing at 1750 F. and annealing at 1750 F. was found to have a relatively uniform microstructure and hardness as shown in Fig. 5 and Table III.

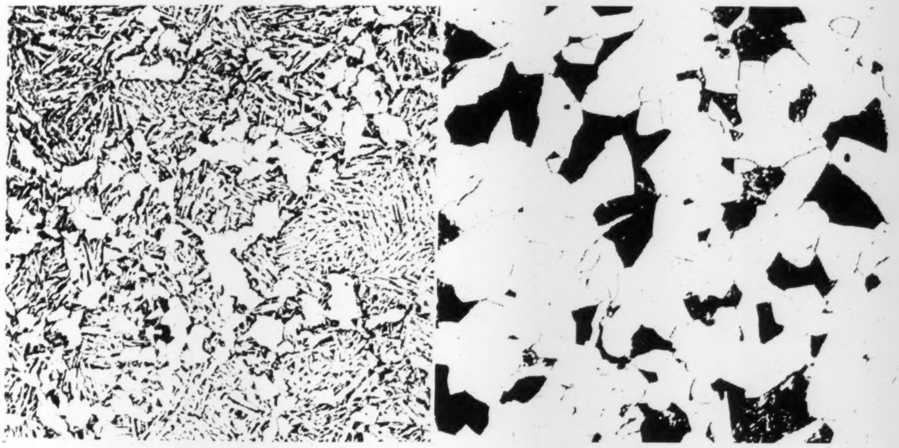

(*a*) Normalized at 1750 F. (*b*) Annealed at 1750 F.

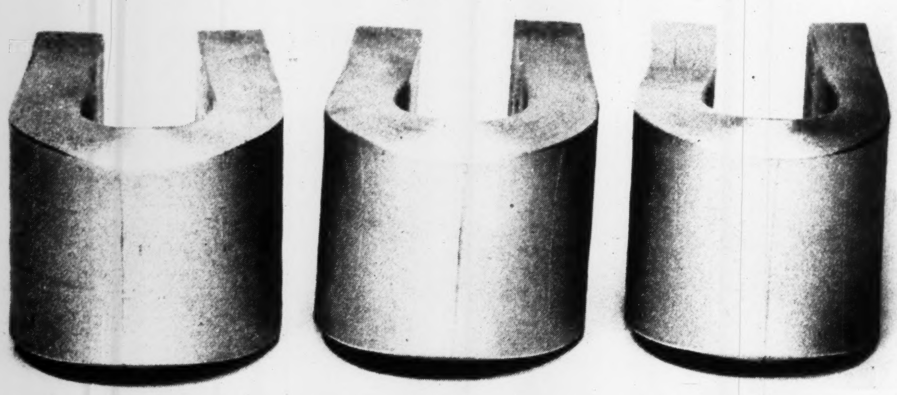
FIG. 5.—12-in., 160 Schedule Pipe, As-Bent Condition (× 100).

The physical properties of the pseudo-bent pipe as obtained from 0.505-in. longitudinal tension specimens removed from the wall of the pipe both before and after heat treatment are also given in Table III.

It is to be noted that the specimens pulled in the as-bent condition have a remarkably high yield and ultimate strength with relatively good ductility. The quenching of pipe during hot bending is in reality a modification of the Austempering process, that is, rapidly quenching to a medium to high subcritical temperature and allowing transformation to take place at this, or slightly higher temperature. Davenport has pointed out that for a given tensile strength and hardness, the ductility properties of steel can be improved by the use of the Austempering method as compared with normal methods of heat treatment (5).

All specimens, the results of which are given in Table III, failed with a "cup and cone" type of fracture.

In Fig. 6 are shown specimens which were removed longitudinally from the 12-in. pseudo-bent pipe, both before and after heat treatment, after bending to a $\frac{11}{16}$-in. radius. The percentage outside fiber elongation in 1 in. is also given in this figure.



(*a*) As-bent condition, 40.8 per cent elongation.
(*b*) As-bent condition, plus normalized at 1750 F., 43.1 per cent elongation.
(*c*) As-bent condition, plus annealed at 1750 F., 44.9 per cent elongation.

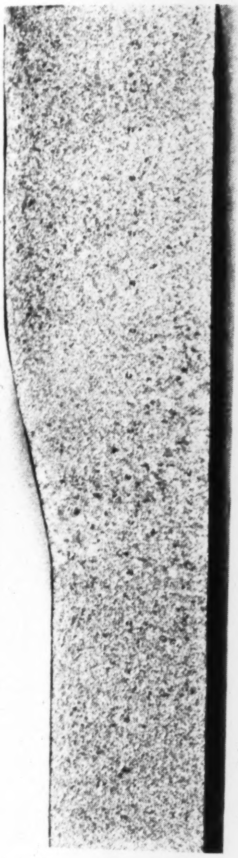
FIG. 6.—Longitudinal Bend Tests Taken from Wall of 12-in., 160 Schedule Pipe (× 1).

It is to be observed that the ductility of the specimen in the as-bent condition is surprisingly high. From Fig. 6 indications are that all three specimens could no doubt have been completely flattened without failure.

Upsetting:

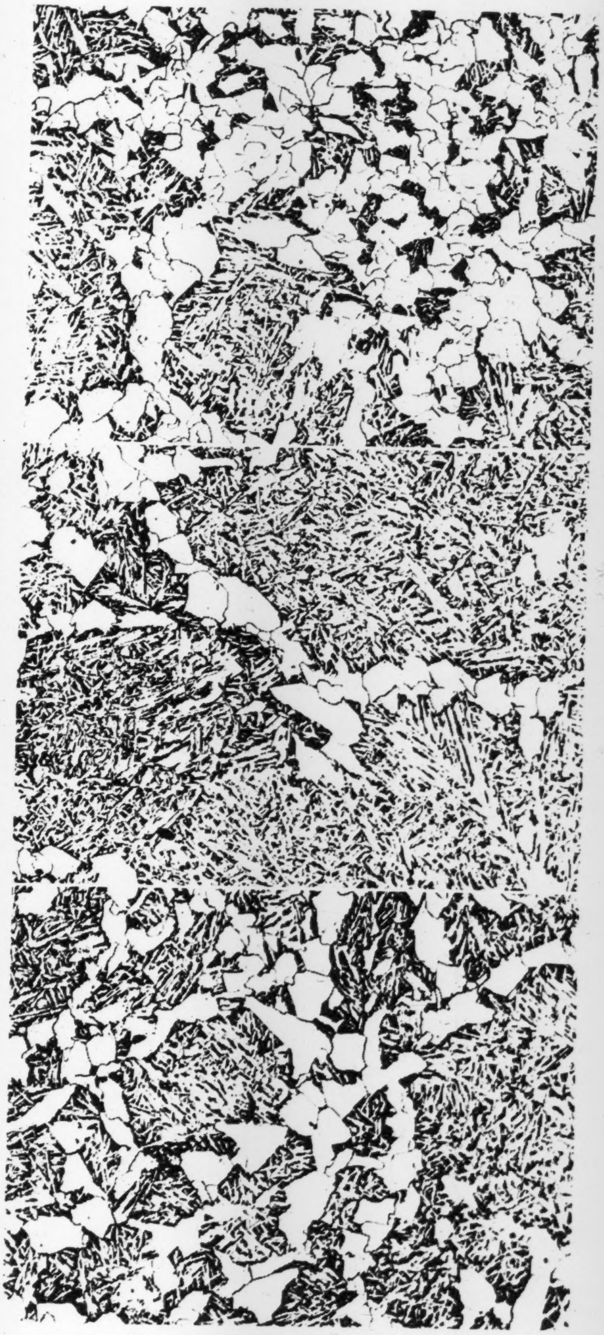
Upset pipe ends have been, and are still used for the dual purpose of providing additional weld metal thickness at the pipe joint and also to provide for a completely recessed backing ring, thus eliminating any possible restriction to fluid flow. A completely recessed backing ring is always to be desired but the need for additional weld metal thickness over the nominal $\frac{1}{16}$ to $\frac{1}{8}$-in. reinforcement is gradually being considered as unnecessary.

In the production of an upset pipe end, the pipe end is placed in a furnace and heated to 1800 to 2100 F. The pipe is then clamped in a die which is recessed to allow for the thickening of the pipe end. A second revolving die is then placed within and against the end of the pipe, producing a combined internal rolling and upsetting operation. Two heatings are usually required to complete an upset. The cooling conditions imposed upon the upset are only those of the upsetting dies and the surrounding air. Since the upset is usually finished at a red heat and then air cooled the microstructure of the upset is that of a Widmanstaetten carbide structure. It is to be observed from Fig. 7 that a



(a) (b) (c)

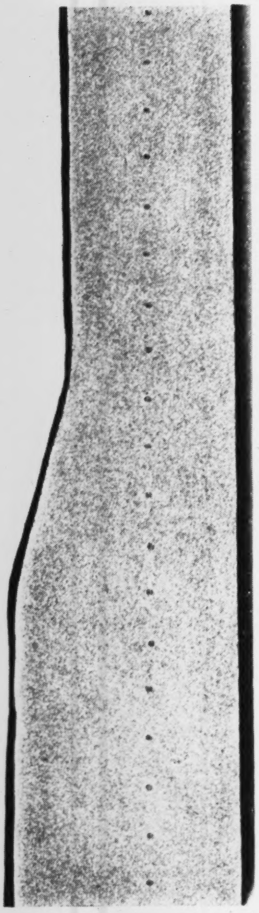
Etched in 20 per cent cold aqueous nitric acid (× 1).



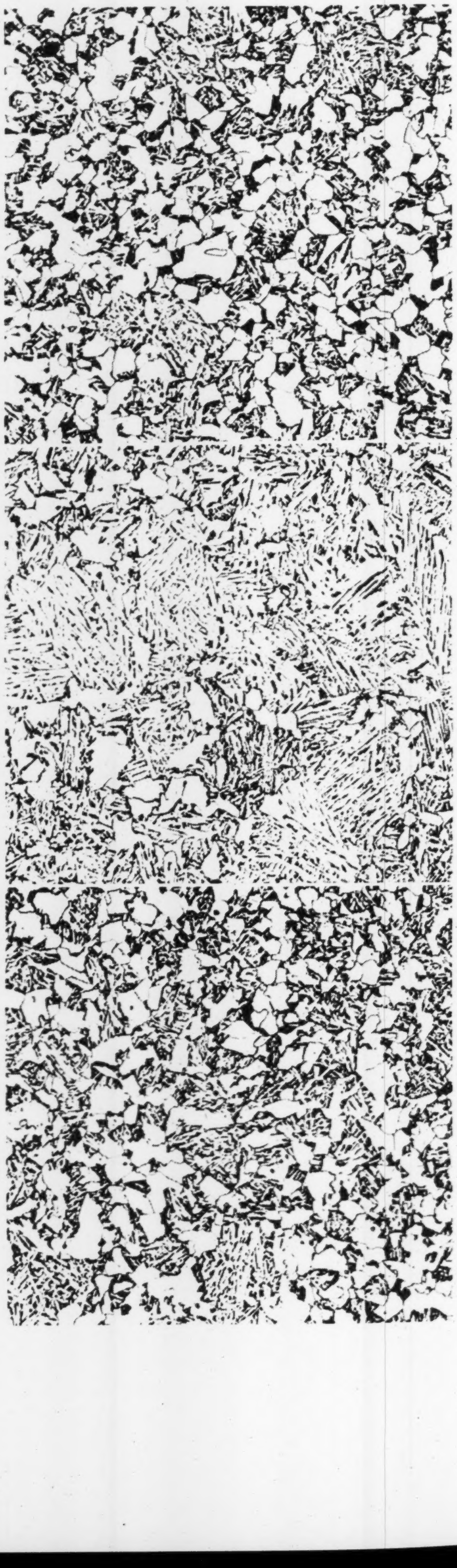
(a) (b) (c)

Etched in saturated picral (× 100).

FIG. 7.—Macro and Microstructure of Upset Pipe End in As-Upset Condition.

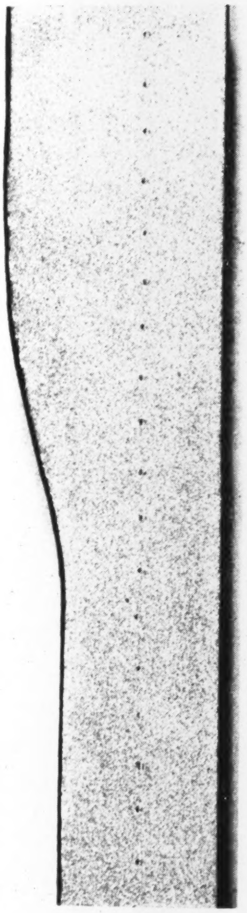


Etched in 20 per cent cold aqueous nitric acid (× 1).

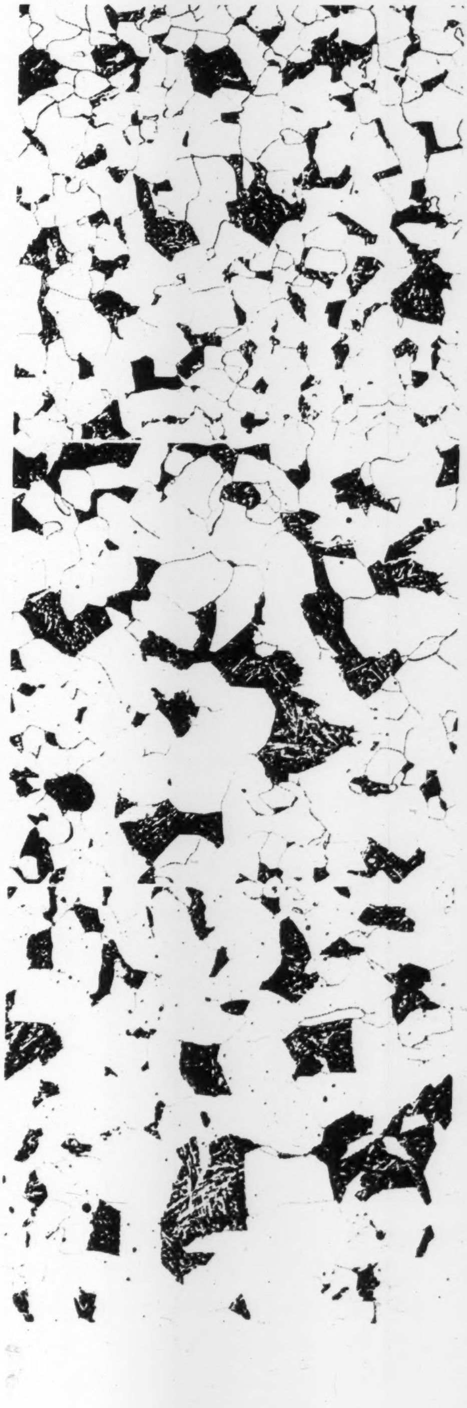


Etched in saturated picral (× 100).

FIG. 8.—Macro and Microstructure of Upset Pipe End After 1750 F. Normalize.



Etched in 20 per cent cold aqueous acid (× 1).

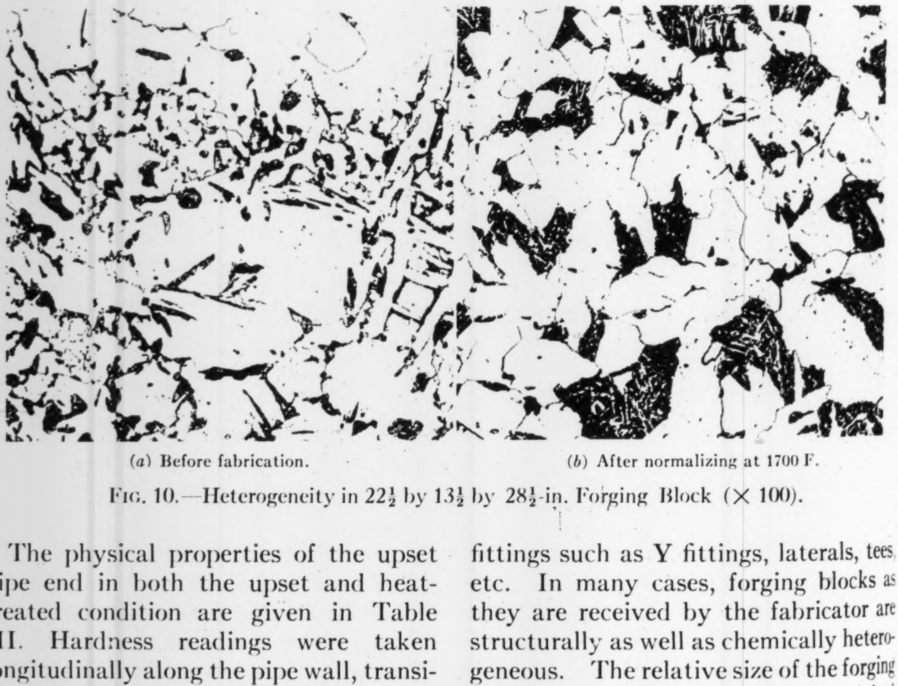


Etched in saturated picral (× 100).

FIG. 9.—Macro and Microstructure of Upset Pipe End After 1750 F. Anneal.

coarse-grained band exists in the transition between the pipe and upset. This is more clearly seen from the accompanying microstructures of Fig. 7.

Figures 8 and 9 show respectively the effect of normalizing at 1750 F. and annealing at 1750 F. on both the macro- and microstructures of the upset end. It is to be observed from the macrostructures of both Figs. 8 and 9 that the coarse-grained transition zone appearing in Fig. 7 has been effectively removed by heat treatment. The accompanying microstructures of Figs. 8 and 9, however, still indicate minor variations.



(*a*) Before fabrication.

(*b*) After normalizing at 1700 F.

FIG. 10.—Heterogeneity in $22\frac{1}{2}$ by $13\frac{1}{2}$ by $28\frac{1}{2}$-in. Forging Block (× 100).

The physical properties of the upset pipe end in both the upset and heat-treated condition are given in Table III. Hardness readings were taken longitudinally along the pipe wall, transition zone, and upset end, the indentations from which may be seen in Figs. 7 to 9. The hardness was found to be relatively uniform for each condition of heat treatment. Tension specimens were machined from $\frac{7}{8}$ in. thick pieces, having the same longitudinal cross-section as that of Fig. 7. All physical properties were raised somewhat over those in the as-upset condition by normalizing at 1750 F. In the case of both the upset and hot-bent pipe, 30 per cent elongation in 2 in. was obtained, however, only from the annealed specimens.

THE EFFECT OF FINISHING TEMPERATURE AND PERCENTAGE OF HOT REDUCTION ON THE GRAIN SIZE AND CARBIDE STRUCTURE OF FORGED CARBON-MOLYBDENUM STEEL

Forgings play an important part in steam power-plant piping by their use in forged and bored steam headers; and fittings such as Y fittings, laterals, tees, etc. In many cases, forging blocks as they are received by the fabricator are structurally as well as chemically heterogeneous. The relative size of the forging block as compared with the original cross-sectional size of the ingot is believed in many instances to result in such structural heterogeneity since the blocks are of such a size that they do not get a sufficient amount of hot reduction. Fig. 10(*a*) illustrates the heterogeneous structure found in a $22\frac{1}{2}$ by $13\frac{1}{2}$ by $28\frac{1}{2}$-

in. forging block prior to fabrication. Indications are, from the microstructure, that the block had been reheated after forging to 1425 to 1475 F. After machining and boring, the fitting was normalized at 1700 F. The microstructure after this final heat treatment was found to have an average grain size of Nos. 4 to 5 as shown in Fig. 10(*b*).

The effect of finishing temperature and percentage of hot reduction on forgings is illustrated in Figs. 11 to 14. An 8 by 8-in. billet section is shown in Fig. 11. As indicated, three distinct zones of varying grain size may be observed. The microstructure of these three zones are shown in Fig. 12 at a magnification of 75 diameters. It may be seen that the grain structure is much too coarse and heterogeneous to be normally considered acceptable for high-temperature service. If, however, it is necessary to use material of the cross-section indicated, a relatively uniform grain can be obtained by soaking at a uniform temperature of 1675 to 1725 F. The effect of forging two 8 by 8-in. sections as shown in Fig. 11, to 3 by 3-in. sections is shown in Fig. 13. In order to effect such a reduction, one reheating was necessary. After reheating, one of the forgings was reduced to final size as rapidly as possible in order to obtain a relatively high finishing temperature.

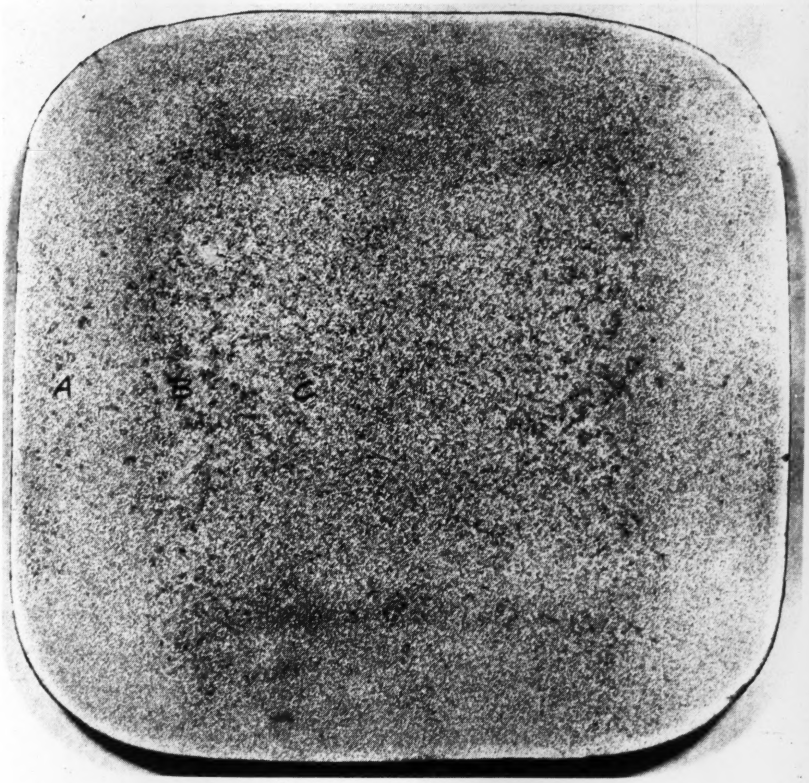
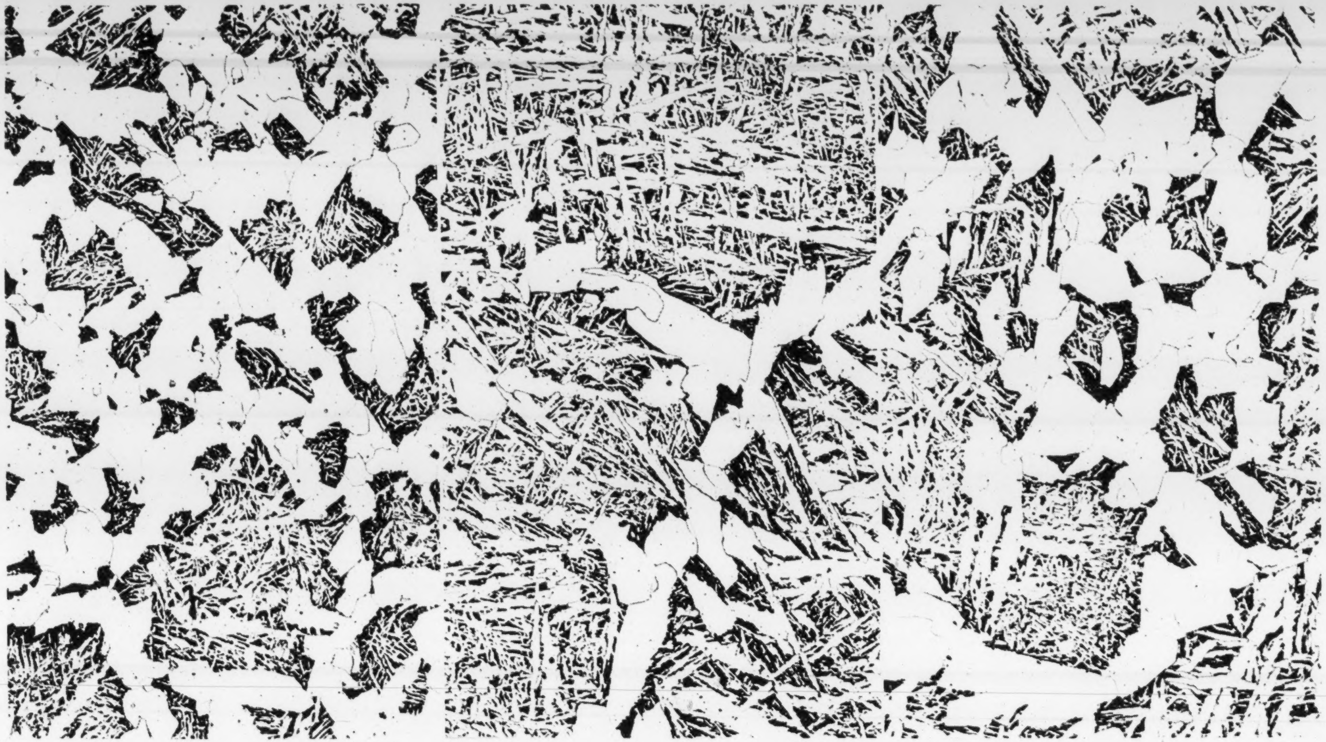


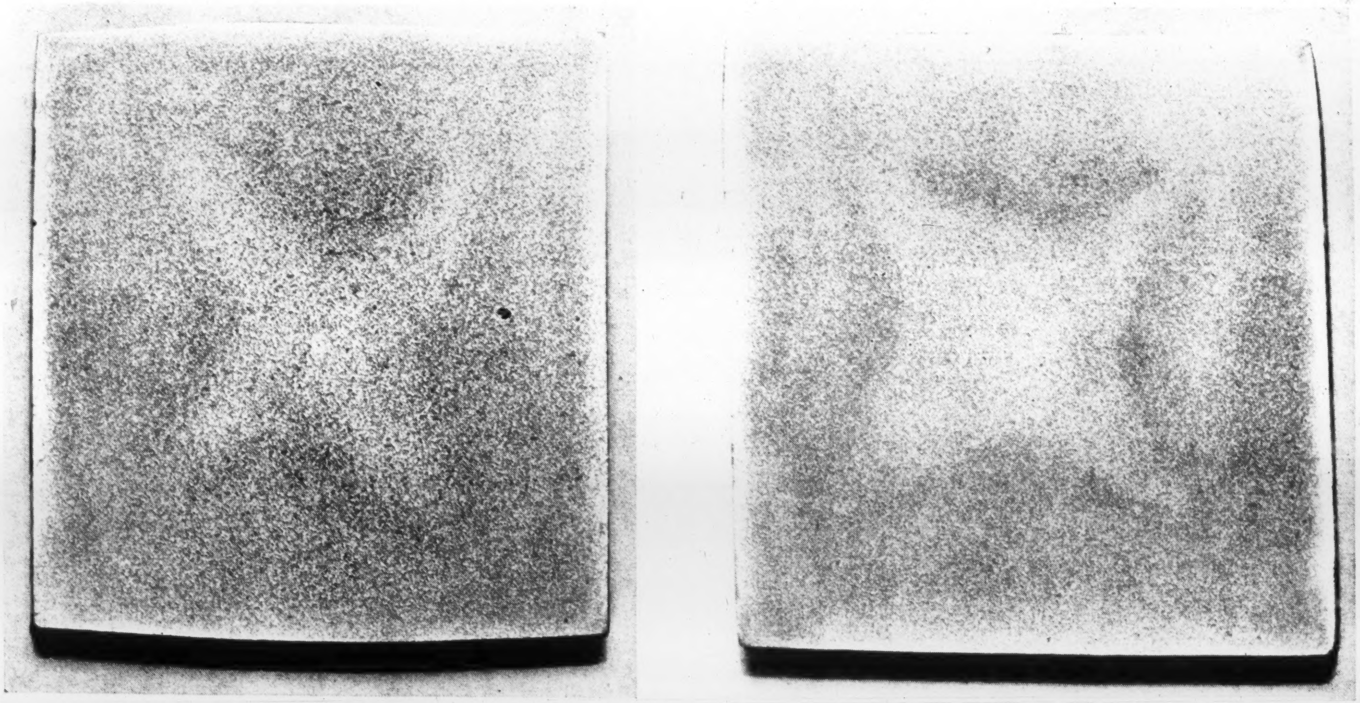
FIG. 11.—Macrostructure of 8 by 8-in. Billet ($\times \frac{1}{2}$).

Etched in 20 per cent cold aqueous nitric acid.



(a) (b) (c)

FIG. 12.—Microstructure of 8 by 8-in. Billet of Fig. 11 (× 75).
Etched in saturated picral.



(*a*) Forging finished at high temperature (approximately 1750 F.).

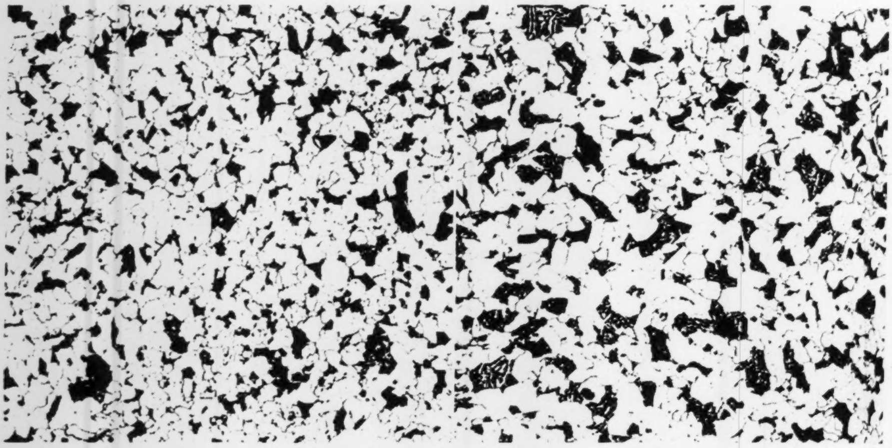
(*b*) Forging finished at low temperature (approximately 1500 F.).

FIG. 13.—3 by 3-in. Sections Forged from 8 by 8-in. Billet.

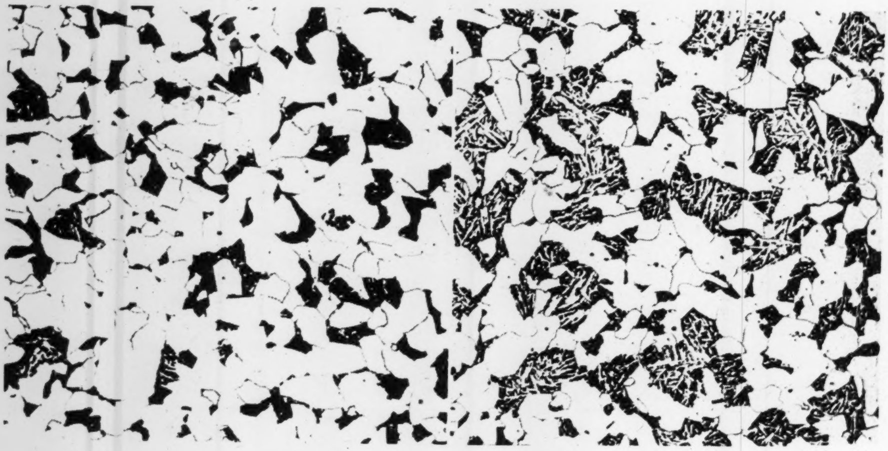
Etched in 20 per cent cold aqueous nitric acid.

The finishing temperature in this case was estimated at 1750 F. The other forging, however, was reduced to 3.5 by 3.5 in. and then allowed to cool to the lowest temperature at which the operator could finish forging to 3 by 3 in. The temperature at which the forging was finished in this case was estimated at 1500 F. The effect of the finishing temperature on the grain size, as shown by Fig. 13 was found to be quite noticeable.

Center of forging. Edge of forging.



Forging finished at low temperature (grain size 6 to 8).

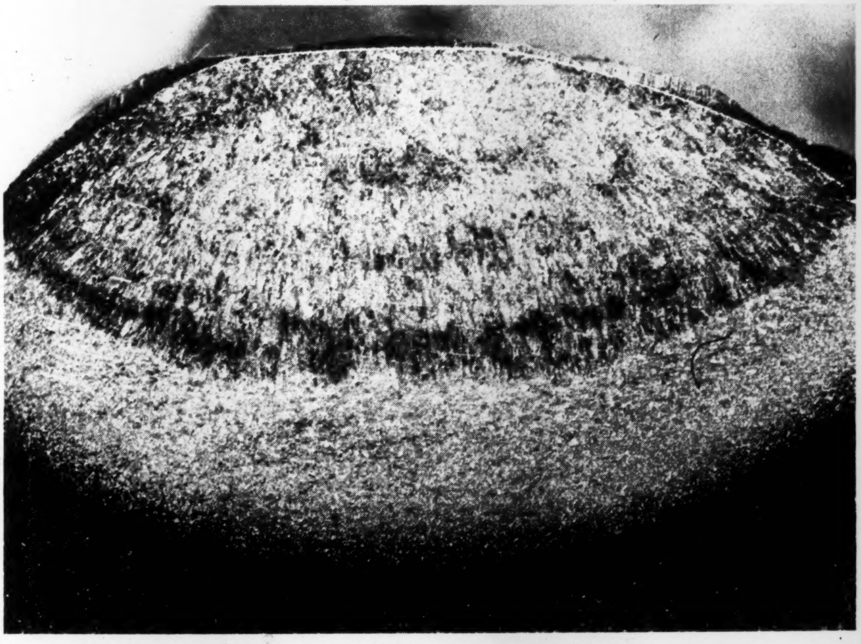


Forging finished at high temperature (grain size 4 to 6).

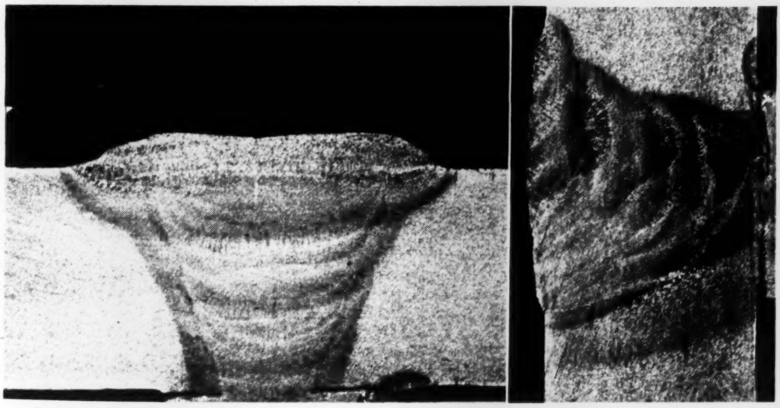
FIG. 14.—Microstructure of Forgings of Fig. 13.

Etched in saturated picral.

The microstructure of the center and edge of the two forgings of Fig. 13, are shown in Fig. 14. The estimated difference of 250 F. in the finishing temperature produced an average difference in grain size of approximately two A.S.T.M. grain size numbers. Another point which was noted was the difference in structure between the center and edge of the forgings. The edge microstructure is typically Widmanstaetten; the microstructure of the center, however, has a dense sorbitic pearlite structure which is abnormal for a normalized structure. Though the center cools somewhat more slowly than the outer edge, it is felt that this change in microstructure occurs abruptly at the edge of the ingot pattern which still persists in the 3 by 3-in.



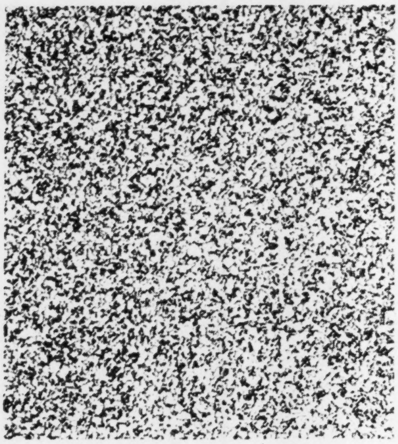
(*a*) Single-bead weld and heat-affected zone (× 6½).



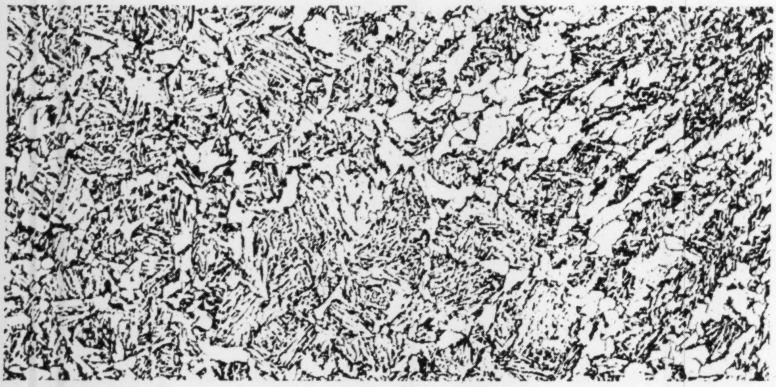
(*b*) Multiple-pass weld, horizontal pipe weld (× 1).

(*c*) Multiple-pass weld, vertical pipe weld (× 1).

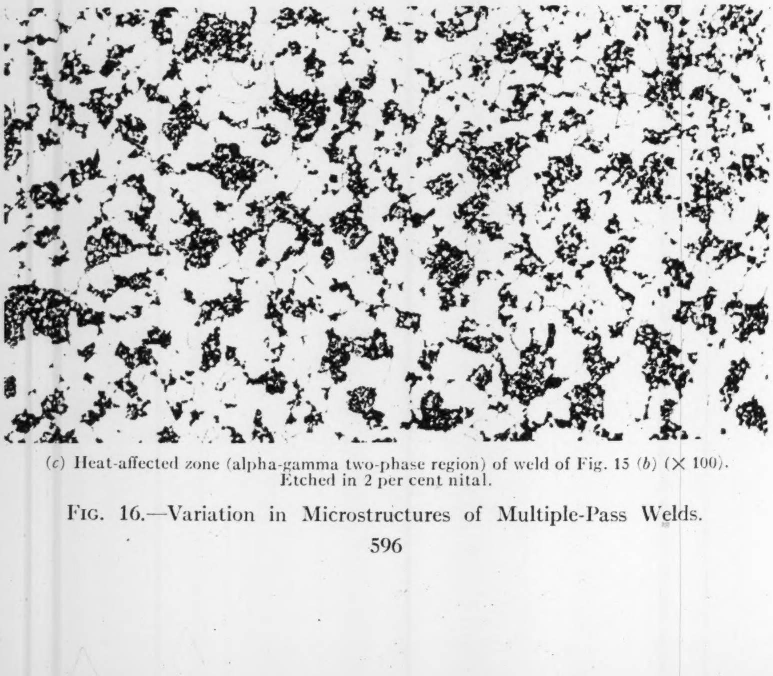
FIG. 15.—Macrostructure of Single-Bead and Multiple-Pass Welds.
Etched in cold aqueous nitric acid.



(*a*) Weld metal of Fig. 15 (*c*) (× 100).
Grain size 9 to 10. Etched in 2 per cent nital.



(*b*) Weld metal, line of fusion, and heat-affected zone of surface bead of Fig. 15 (*b*) (× 100).
Etched in 2 per cent nital.



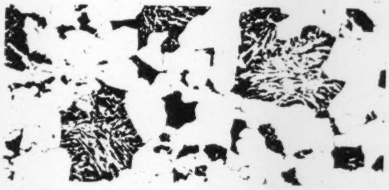
(*c*) Heat-affected zone (alpha-gamma two-phase region) of weld of Fig. 15 (*b*) (× 100).
Etched in 2 per cent nital.

FIG. 16.—Variation in Microstructures of Multiple-Pass Welds.

forgings. There appears to be a similarity between the center microstructures and that of a fine-grain type of steel normalized at 1750 F. as shown in Fig. 18.

MICROSTRUCTURE AND PHYSICAL PROPERTIES OF WELDED JOINTS

Present-day practice in the fabrication of carbon-molybdenum piping for high-temperature service appears to be



Original structure, normalized at 1750 F. Approximate cooling rate 1600 F. per hour.

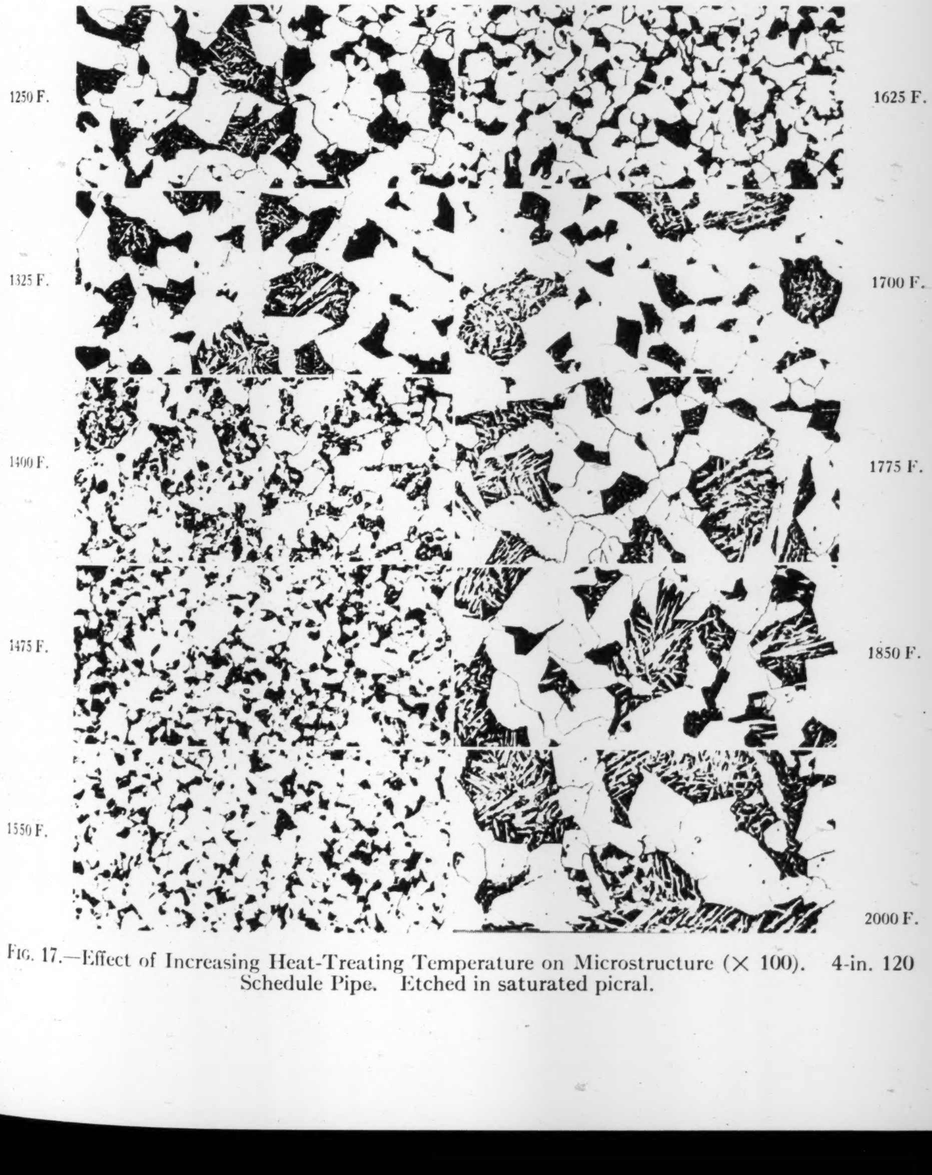


FIG. 17.—Effect of Increasing Heat-Treating Temperature on Microstructure (× 100). 4-in. 120 Schedule Pipe. Etched in saturated picral.

as follows: After all hot working operations such as swadging, upsetting, hot bending, etc., have been completed, the pipe is completely heat treated for grain size restoration. The pipe ends, after heat treatment, are beveled and fitted up for welding. Welding practice consists of preheating the joint to 350 to 500 F. and maintaining this temperature during welding, followed by a 1200 F. stress-relieving treatment. A temperature of 1200 F. has no apparent change on the microstructure of either the weld metal or heat-affected zone of which the welded joint is comprised. Figure 15 illustrates the macrostructure of both a single-bead, and multiple-pass welds The single-bead weld indicates a variety of structures to be present, including the as-cast columnar weld metal, the parent metal which was heated to a sufficiently high temperature to be fully austenitic, and a zone which was heated only to the temperature of the two-phase alpha-gamma region. The multiple-pass horizontal and vertical pipe welds also shown were removed from 8-in., 160 schedule pipe and are characteristic of those found in welded heavy-wall carbon-molybdenum pipe. Variations in microstructure of multiple-pass welds are shown in Fig. 16. Each pass or bead of weld metal as deposited in multiple-pass welds is partially or completely refined by the heat from successive layers of weld metal applied, the last bead or layer deposited being the only one which is completely in the as-cast or as-deposited state. Refined weld metal is always exceedingly fine grained, it being doubtful that the grain size would ever exceed No. 7 and in most cases will be found to be much smaller. The weld metal of Fig. 15(*c*), the microstructure of which is shown in Fig. 16(*a*) has a grain size of Nos. 9 to 10. The weld metal of the surface bead of the horizontal pipe weld of Fig. 15(*b*) and adjacent parent metal, is also shown in Fig. 16(*b*). The weld metal, though columnar has a Widmanstaetten carbide structure as does the adjacent parent metal. This type of structure is generally considered acceptable for high-temperature service. The fine grain structure found in the weld metal, however, if found in pipe would be considered unacceptable. A striking parallel may be seen to exist between Fig. 16(*c*) showing the two-phase region of the heat-affected zone of the multiple pass horizontal weld of Fig. 15(*b*) and specimens which were heated to 1400 to 1550 F., inclusive, the microstructures of which are shown in Fig. 17. This structure (Fig. 16(*c*)) with the present method of acceptance based on grain size, would be considered highly unsatisfactory. It is to be pointed out, however, that all microstructures of Fig. 16 do exist in welded carbon-molybdenum joints in the stress-relieved condition.

TABLE IV.—PHYSICAL PROPERTIES OF WELDED JOINTS IN CARBON-MOLYBDENUM PIPE AFTER STRESS RELIEVING.

Reduced-Section Tensile Strength, psi.	Free Bend Ductility Elongation in Outside Fibers, per cent	Brinell Hardness
63 000 to 67 000 (failure in parent metal)...............	40 to 55	140 to 155

As was previously mentioned, it is not the purpose of this paper to pass judgment on the possible significance of such existing conditions from the standpoint of creep rate, but merely to point out that such conditions do exist.

The room temperature physical properties obtainable from welded joints in heavy-wall carbon-molybdenum pipe in the preheated and stress-relieved condition are given in Table IV. From Table IV it is to be observed that excellent room temperature physical test results are obtainable from welded car-

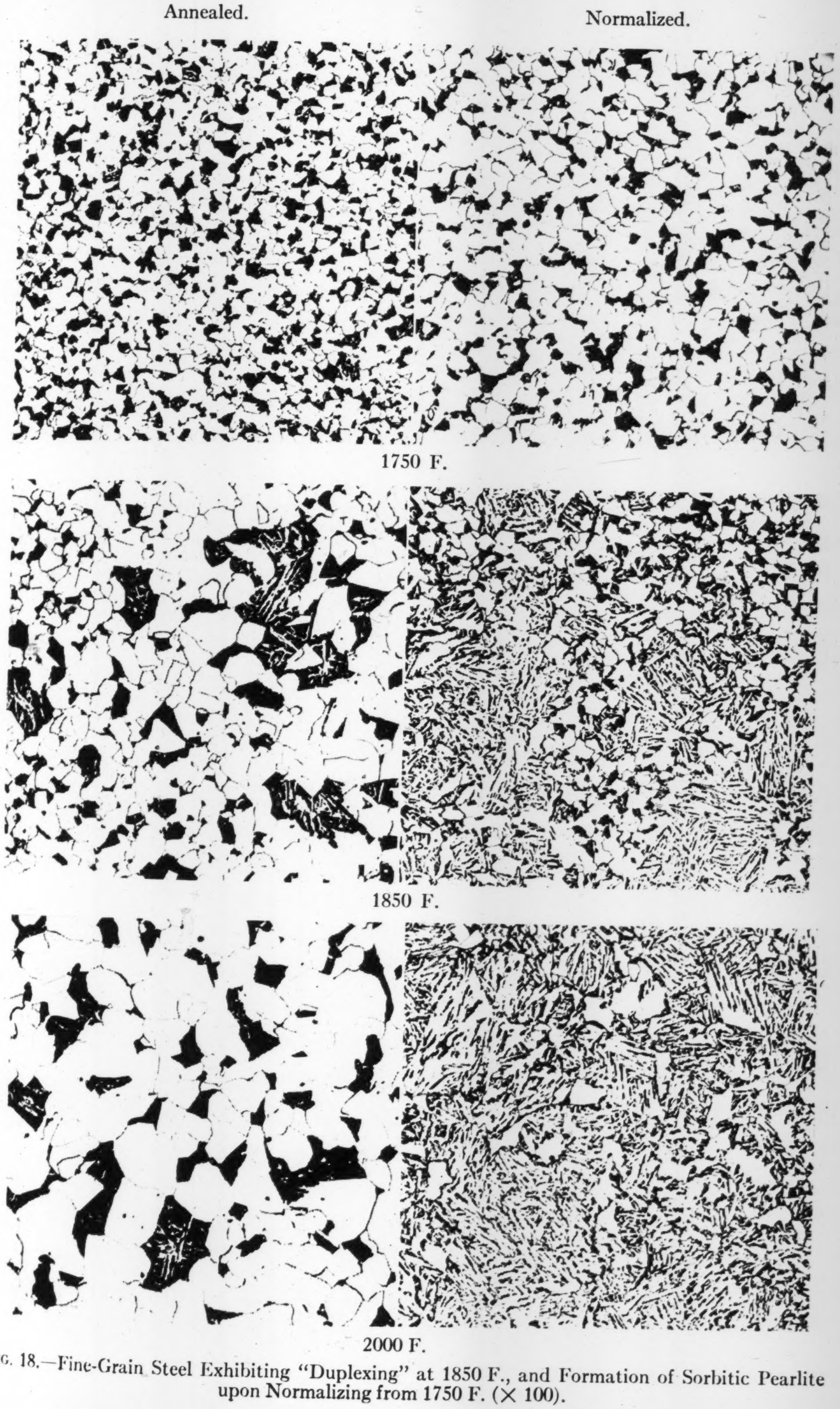

Annealed.
Normalized.
1750 F.
1850 F.
2000 F.

FIG. 18.—Fine-Grain Steel Exhibiting "Duplexing" at 1850 F., and Formation of Sorbitic Pearlite upon Normalizing from 1750 F. (× 100).

bon-molybdenum pipe in the stress-relieved condition.

EFFECT OF HEAT-TREATING TEMPERATURE, COOLING RATE, AND STEEL MELTING PRACTICE ON FINAL GRAIN SIZE AND CARBIDE STRUCTURE

Grain size and carbide structure are essentially dependent on heat-treating temperature, cooling rate, and steel melting practice since improper control of any one of these factors may give rise to rejection of pipe for service conditions of 900 F. or higher.

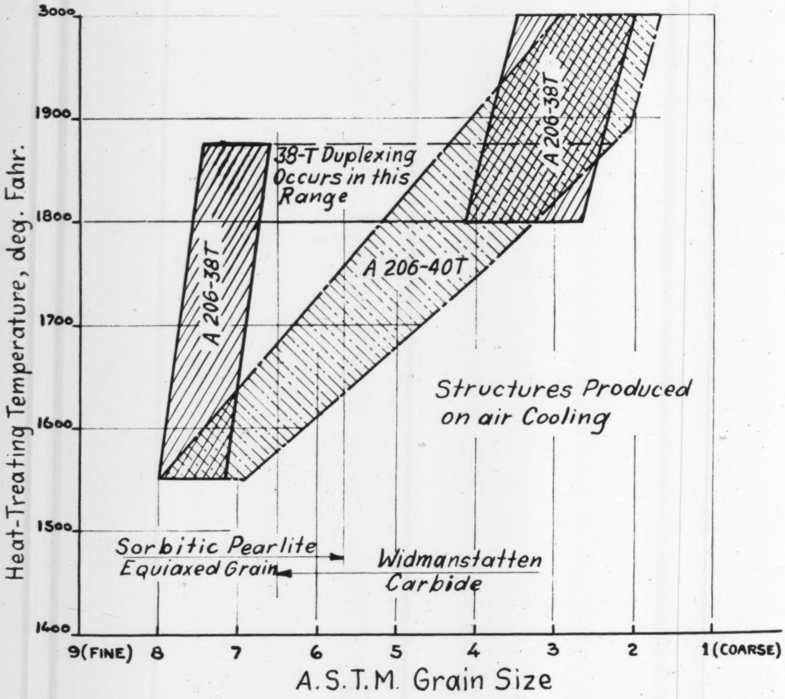


FIG. 19.—Schematic Variation in Grain Size and Carbide Structure as a Function of the Heat-Treating Temperature.

Carbon-molybdenum steel A 206.
Fine grain A 206 – 38 T.
Coarse grain A 206 – 40 T.

The effect of increasing heat-treating temperature on microstructure is illustrated in Fig. 17, other conditions remaining constant. A 1-in. wide ring of 4-in., 120 schedule pipe was heated to 1750 F. and cooled approximately 1600 F. per hr. This ring was then cut into 1-in. specimens and reheated to temperatures from 1250 to 2000 F. in steps of 75 F. followed by cooling at the above-mentioned rate. The material used was taken from 4-in. pipe, heat No. 2, Table I.

It is to be observed from Fig. 17 that the microstructure resulting from the previous 1750 F. normalize, continues to exist through 1325 F. A continual change in microstructure occurs from 1400 through 1550 F., the temperature range at which the steel undergoes transformation. From 1625 to 2000 F., no particular change occurs other than grain coarsening, except that as the grain size increases, the pearlite areas become less dense, 1700 F. and above exhibiting a Widmanstaetten carbide structure.

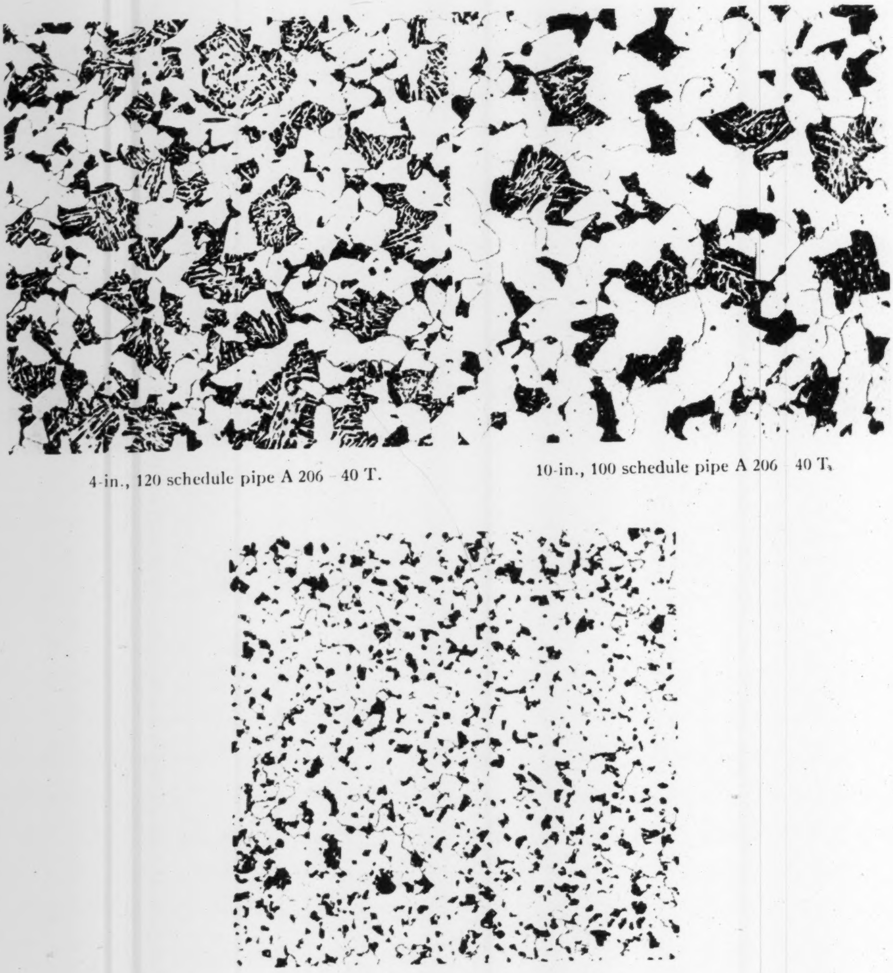
Photomicrographs of specimens removed from a 10-in., 100 schedule pipe exhibiting fine-grain characteristics (A 206 – 38 T) are shown in Fig. 18. Air cooling (normalizing) this material from 1750 F., resulted in a No. 7 grain size with the carbide present as dense patches of sorbitic pearlite normally characteristic of a slow-cooled material. Definite duplexing tendencies were observed in this material when heated to 1850 F. followed by both furnace and air cooling. The air-cooled specimens exhibited patches of both coarse-grained Widmanstaetten carbide and fine-grained ferrite and sorbitic pearlite. The slow-cooled specimens exhibit both coarse and fine grain size, essentially all carbide, however, being in the form of sorbitic pearlite. The structures, normalized and annealed from 2000 F., are equivalent to those normally obtained by respective cooling from 1700 to 1750 F. with A 206 – 40 T material.

A coarse-grained material made in accordance with Specifications A 206 – 40 T will usually be found to coarsen gradually with increasing temperature, though in most cases the extent of the coarsening is slightly above 1800 F. unless the time at temperature is unduly prolonged. A fine-grained material made in accordance with Specifications A 206 – 38 T will usually be found to remain fine up to 1800 to 1875 F. at which range coarsening occurs in patches, as shown in Fig. 18. This condition is called duplexing. Further heating to 1925 to 2000 F. will normally result in coarsening of the entire structure. Figure 18 illustrates the fact that a normally fine-grain steel can be made to coarsen by using excessively high heat-treating temperatures. From the customer's standpoint, however, it is desirable to obtain the desired grain structure at the lowest possible heat-treating temperature since oxidation (scaling) may possibly encroach on the minimum wall thickness of the pipe when excessive heat-treating temperatures are used. The variation in grain size and carbide structure as a function of heat-treating temperature is shown schematically in Fig. 19, including the approximate spread in grain size for any one heat-treating temperature.

Proper ferrite grain size and carbide structure, of which good creep properties in carbon-molybdenum steel appear to be dependent, for a given cooling rate are dependent to a large extent on the austenite grain size, which is in turn dependent on the maximum heat-treating temperature. Maximum heat-treating temperature and cooling rate to produce a given austenite grain size, and subsequent ferrite grain size and carbide structure, is to a very great extent dependent on the amount of aluminum added to the steel.

Take for example a carbon-molybdenum steel containing $\frac{1}{2}$ lb. of aluminum per ton. Upon heating this steel to 1700 to 1750 F. an austenitic grain size should be established which will normally produce upon air cooling a No. 4 to No. 6 Widmanstaetten carbide-ferrite grain size. Air cooling is sufficiently fast so that complete rejection of the ferrite to the austenite grain boundaries is impossible while cooling through the transformation range of the steel, resulting in somewhat of an acicular (Widmanstaetten) carbide grain structure (*6*).

In contrast to this, a carbon-molybdenum steel containing 2.25 lb. of aluminum per ton, when heated to 1700 to 1750 F. would establish a fine grain size of Nos. 7 to 8. Since the austenitic grain size is small, the distance from center to edge of the austenitic grain is also small and for the same cooling rate as in the previous example, sufficient time elapses for essentially complete rejection of the proeutectoid ferrite since it has a lesser distance to travel to the austenitic grain boundary, with the result of a sorbitic pearlite and an equiaxed grain structure. Thus aluminum is believed to have an indirect affect on the creep characteristics of carbon-molybdenum steel by affecting the grain size and carbide structure of the material for a given heat-treating cycle. The effect of aluminum on microstructure is further substantiated by comparison of photomicrographs shown in Fig. 20. Two of the steels, the microstructure of which is shown, were made in accordance with Specifications A 206 - 40 T; the third was made in accordance with Specifications A 206 - 38 T. The microstructures shown in



4-in., 120 schedule pipe A 206 - 40 T.

10-in., 100 schedule pipe A 206 - 40 T.

10-in., 100 schedule pipe A 206 - 38 T

FIG. 20.—Production Heat Treatment, 1700-1725 F., Normalized (× 100).

Fig. 20 were obtained from pipe coupons placed in a production heat treatment of pipe and normalized at 1700 to 1725 F. It is to be observed that a variation in density of the pearlite (or carbide) exists between the two structures exhibiting the Widmanstaetten pattern. This is due to a difference in cooling rate resulting from a difference in pipe wall thickness.

SUMMARY

The present method of acceptance of carbon-molybdenum piping for high-temperature service is based on metallographic examination for grain size and structure.

Though there appears to be general agreement as to the proper grain size for increased creep strength, there also appears to be disagreement as to the rate of cooling of pipe from the heat-treating temperature.

On the basis of both metallographic and hardness examinations, carbon-molybdenum steel has a relatively rapid transformation rate.

Water quenching during hot bending produced an increase in both the yield and tensile strengths of this material with little loss in ductility. Good ductility is insured because of slow cooling from 1000 to 800 F. to room temperature resulting from the contained heat of the sand within the pipe.

An increase in both yield and tensile strengths with good ductility also resulted from hot working during the upsetting of pipe ends.

Change in microstructure and physical properties of pipe resulting from hot fabrication is from all indications completely restored after heat treating for proper grain size.

Increasing amounts of hot reduction by forging, in carbon-molybdenum steel remarkably improves any existing structural heterogeneity. The temperature at which the forging operation is discontinued determines its grain size. Carbon-molybdenum forgings exhibiting structural heterogeneity, but which because of their dimensions cannot be further hot worked, can be greatly improved by a thorough soak at the requisite heat-treating temperature and air cooled from that temperature.

Heat treating to establish the proper grain size is done prior to welding. The heat from the welding arc results in a narrow band on either side of the weld, to be heated into and through the transformation range of the heat-treated pipe. The microstructure of both the heat-affected zone of the weld and the deposited weld metal would be considered as unsatisfactory if found in the pipe proper. The questions are asked: "What are the creep characteristics of the heat-affected zone of a weld made on carbon-molybdenum steel? How does the fact that fine-grained weld metal has been found to have relatively good creep properties fit into the picture of the necessity of a medium coarse grain with a Widmanstaetten carbide for good creep properties?"

Aluminum additions to carbon-molybdenum steel are believed to indirectly affect the creep properties of this material by influencing the austenitic grain size, which in turn influences the ferrite grain size and carbide structure for a given heat-treating cycle.

Acknowledgments.

The author wishes to acknowledge the helpful suggestions and constructive criticism given by G. S. Larsen, Chief Engineer, and G. W. Petrie, Jr., Plant Superintendent, Pittsburgh Piping and Equipment Co.

The author is grateful for the laboratory assistance given by Robert Geisler.

The author wishes to express appreciation to others within the organization whose assistance made this work possible.

The 12-in., 160 schedule pipe and the 8 by 8-in. billet section were furnished through the courtesy of E. C. Wright, Chief Metallurgist, National Tube Co.

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(1) S. H. Weaver, "Actual Grain Size Related to Creep Strength of Steels at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 176 (1938).

(2) H. C. Cross and J. G. Lowther, "Study of Effects of Variables on the Creep Resistance of Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 125 (1940).

(3) S. H. Weaver, "Relation of Grain Size to Creep Strength of Carbon-Molybdenum Steel," *General Electric Review*, Vol. 43, No. 9, p. 357 (1940).

(4) A. E. White and Sabin Crocker, "Effect of Grain Size and Structure on Carbon-Molybdenum Steel Pipe," *Preprint No. 32*, Am. Soc. Mechanical Engrs., December, 1940.

(5) E. S. Davenport, "The Process and Result of Austenite Transformation at Constant Temperature," *Metals and Alloys*, January, 1937.

(6) J. R. Vilella, "The Grain Size of Steel," *Mechanical Engineering*, April, 1940, p. 293

DISCUSSION

Mr. Sabin Crocker[1] *(presented in written form).*—Mr. Emerson's paper is of particular value in that it adds to the available fund of information about this relatively new product for which a background of factual data, metallographic appearance, and performance should be built up as rapidly as possible. It is decidedly helpful to the users of carbon-molybdenum pipe to have references available which serve to identify grain size and structure with heat treatment for specimens of this type of steel which have been made by different melting practices or heat treated in different ways, either intentionally, or incidentally during fabrication. Also it is helpful to have data on the characteristics of forged material and of welds in carbon-molybdenum pipe.

It is gratifying to note that many of the author's findings tend to confirm and better establish the identification of certain types of grain size and structure in carbon-molybdenum pipe steel with respective melting practices and heat treatments as noted in the paper by White and Crocker presented before The American Society of Mechanical Engineers last December.[2] While Mr. Emerson's results do not embrace the behavior of specimens under conditions of high-temperature service, this association of metallographic appearance with case history of the steel is useful in that the structural characteristics of the grains serve as thumbprint patterns linking the antecedents of a given steel with the behavior to be expected of it in service. Thus, where the antecedents of some lot of pipe are unknown, a way is provided for estimating probable behavior from the appearance of the microstructure.

It is reassuring to learn through the photomicrographs of Figs. 1 to 5 of the paper that the grain structure of pipe which was quenched during the process of fabrication can be restored to a satisfactory condition through subsequent heat treatment. This tends to confirm results shown in Figs. 11, 12, and 17 of the White and Crocker paper.

Mr. Emerson asks the following questions concerning the weld structure shown in his Figs. 15 and 16: (*a*) "What are the creep characteristics of the heat-affected zone of a weld made on carbon-molybdenum steel?" and (*b*) "How does the fact that fine-grained weld metal has been found to have relatively good creep properties fit into the picture of the necessity of a medium coarse grain with a Widmanstaetten carbide for good creep properties?"

With reference to (*a*), while the writer's company has not made creep tests from specimens taken across the weld and through the heat-affected zone for carbon-molybdenum pipe, it might be of interest to mention that the Detroit Edison Co. did make such tests several years ago when we first took up welding *carbon* steel pipe for high-pressure service. The results were so satisfactory that we have not been much concerned on this score since and did not undertake to investigate this point for carbon-*molybdenum* pipe. Furthermore, across the weld specimens are neither "fish, flesh nor fowl" and we are not disposed to set much store by them anyway. We did think it worth while, however, to investigate one specimen of "all-weld" metal from a joint in carbon-molybdenum pipe as reported on pages 8 and 9 of the White and Crocker paper.[2] Despite the fine grain of the "all-weld" specimen, it had surprisingly good creep strength at 925 F. and excellent physical properties at room temperature.

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[1] Senior Engineer, Engineering Division, The Detroit Edison Co., Detroit, Mich.

[2] A. E. White and Sabin Crocker, "Effect of Grain Size and Structure on Carbon-Molybdenum Steel Pipe," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 63, No. 8, November, 1941, p. 749.

With reference to (*b*) it would seem that:

1. In the case of circumferential welds, a lower creep strength can be tolerated than in the pipe proper because of the support offered by the adjacent higher strength pipe sections. As far as circumferential or diametral increase is concerned, a weld of only half pipe thickness would not begin to creep as much as the same reduced thickness in a long tube. This point may be verified by examining the tubular creep test data presented in the Van Duzer-McCutchan paper.[3] The thin-walled tube adjacent to the heavier end sections was restrained from bulging by the adjoining pipe. This effect gradually decreased with distance from the ends until at 3 in. no restraint was evident.

2. The total combined longitudinal stress due to bending and longitudinal pressure can be limited at welded joints. Our practice is to limit this stress to 12,000 psi. at welded joints as compared with 15,000 psi. allowed in straight pipe or bends remote from joints.

Mr. Emerson's Fig. 17 showing effect of increasing heat-treating temperature on microstructure serves to show differences produced by 75 deg. Fahr. steps in normalizing temperature. The change in grain size and appearance of microstructure between steps is pronounced, particularly around the critical temperature, and points the need for careful control of furnace temperature if consistent results are to be obtained commercially.

Some similar work presented in Figs. 33 to 40 inclusive of the closure to the White and Crocker paper[2] may be of interest in confirming the effect of steps in the heat-treating temperature. In this case also several specimens from the same piece of pipe were heat treated in different ways and at successively higher temperatures in order to investigate the variation in impact strength as well as in grain size and structure. As can be seen on reference to that paper, the photomicrographs correspond closely to the respective treatments shown in Mr. Emerson's Fig. 17. Since no impact values are furnished in the present paper it may be of interest to repeat the following results from the White and Crocker closure:

RESULTS OF IMPACT TESTS
12-IN. SCHEDULE 100 CARBON-MOLYBDENUM PIPE 268, HEAT 5085

Specimen	Condition of Specimens	Impact Strength ft-lb.[b]	
		Longitudinal	Transverse
No. 1	As received[a]	10	12
No. 2	Drawn at 1200 F.	14	13
No. 3	Annealed at 1650 F.	14	14
No. 4	Normalized at 1600 F.	67	62
No. 5	Normalized at 1650 F.	83	60
No. 6	Normalized at 1700 F.	68	52
No. 7	Normalized at 1800 F.	40	29
No. 8	Normalized at 1650 F. and drawn at 1200 F.	79	63

[a] According to Section 4(*a*) of the Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206-40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 252.

[b] Average of 3 tests—Standard A.S.T.M. Charpy "V" notch specimens.

It should be mentioned that the impact strength of this particular piece of pipe was somewhat below par in the "as received" condition, so that the effect of favorable heat treatment was distinctly marked. From rather limited observation the usual impact strength in the "as received" condition of carbon-molybdenum pipe made by the melting practice conducive to grain coarsening at 1700 F. seems to be of the order of 20 to 40 ft-lb. The likelihood of an occasional piece having an impact strength of only 10 to 12 ft-lb. points to the possible desirability of normalizing some straight pieces as well as bends. With the exception of one heat of steel it has been found possible in the pipe so far examined to secure satisfactory impact properties by a simple normalize.

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[3] R. M. Van Duzer, Jr., and Arthur McCutchan, "High-Temperature-Steam Experience at Detroit," *Transactions*, Am. Soc. Mechanical Engrs., July, 1939, Vol. 61, No. 5, p. 383.

It might be added that the significance of impact values for pipe steel is a debatable question because no one seems prepared to say what value of impact strength should be expected for any particular steel or type of notch. Some persons prefer the A.S.T.M. standard V-notch, while others, including the pipe mill men, are inclined to use the keyhole notch or a simplified version thereof. Results from different varieties of notches do not agree too closely, and sometimes the effect of small flaws in the coupons or of errors in machining the notches will upset the results. But aside from these points, the question has been raised as to what actual indication of the probable behavior of the material in service can be deduced from its impact strength. Some people contend that the property determined with the standard A.S.T.M. V-notch should be termed "notch toughness" rather than "impact strength," and question what significance "notch toughness" has where there is no such notch in the pipe as actually used. The writer appreciates that these remarks about impact strength are outside the scope of Mr. Emerson's paper, but nevertheless offers them in the hope that they may stimulate further thought along these lines.

MR. R. W. EMERSON[4] (*author's closure, by letter*).—Mr. Crocker's discussion is a valuable contribution to the author's findings. With respect to the discussion on impact properties, it is felt that, in view of the rather wide variation in impact strength obtained by variation in heat treatment found and reported in both the White and Crocker paper[2] and the Weaver paper,[5] this property in carbon-molybdenum steel should certainly not be overlooked and further information on this subject would be highly beneficial.

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[4] Metallurgist, Pittsburgh Piping and Equipment Co., Pittsburgh, Pa.

[5] S. H. Weaver, "The Effect of Carbide Spheroidization upon the Creep Strength of Carbon-Molybdenum Steel," see p. 608.

THE EFFECT OF CARBIDE SPHEROIDIZATION UPON THE CREEP STRENGTH OF CARBON-MOLYBDENUM STEEL

By S. H. Weaver[1]

Synopsis

Design stresses and the service life of high-temperature equipment are affected by the progressive changes in the properties of a steel maintained at constant stress and temperature. Creep test data were obtained from a plate of carbon-molybdenum steel in twelve different conditions according to heat treatment and carbide spheroidization. Each condition of the steel was run in two independent long-time creep tests, one at 900 F. and one at 1000 F. and the creep strengths with the resulting physical properties are reported. Data on the time for carbide spheroidization are discussed and tentative conclusions upon the changing properties of the steel during the service life are presented. The strength at 1000 F. is near a practical limit in design stress for in two years service the creep stress has decreased to 50 per cent and in twenty years service to as low as 23 per cent of the initial creep stress. There is a tendency for all of the conditions of the steel to approach an equal stable value.

The object of the creep tests presented in this paper was to determine the service life of high-temperature apparatus, particularly steam turbines. Operating temperatures are gradually being increased and are now approaching values which produce slow changes in the properties of the steel during the service life of equipment. The engineer must know and provide in the initial design for these changed properties. The spheroidization of the carbides in steel produces a weakening effect during the service life of equipment for use at higher temperatures. At present the spheroidizing effect on different steels produced by temperature without stress is known. But before final interpretation of creep results in terms of service

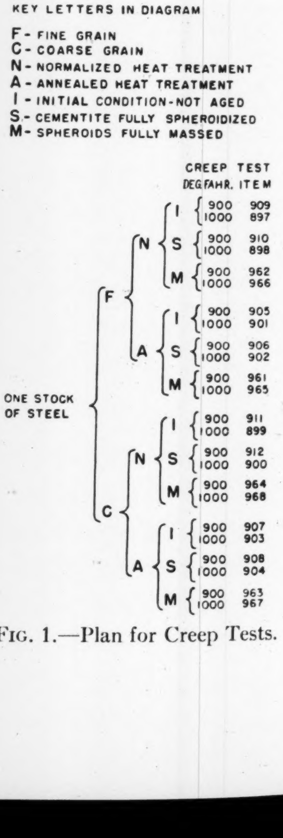
KEY LETTERS IN DIAGRAM
F - FINE GRAIN
C - COARSE GRAIN
N - NORMALIZED HEAT TREATMENT
A - ANNEALED HEAT TREATMENT
I - INITIAL CONDITION-NOT AGED
S - CEMENTITE FULLY SPHEROIDIZED
M - SPHEROIDS FULLY MASSED
CREEP TEST
DEG FAHR.
ITEM
ONE STOCK OF STEEL
F
N
I 900 909
1000 897
S 900 910
1000 898
M 900 962
1000 966
A
I 900 905
1000 901
S 900 906
1000 902
M 900 961
1000 965
C
N
I 900 911
1000 899
S 900 912
1000 900
M 900 964
1000 968
A
I 900 907
1000 903
S 900 908
1000 904
M 900 963
1000 967

Fig. 1.—Plan for Creep Tests.

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[1] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

life can be made it will be necessary to determine the combined effect of temperature and stress. This will require several years of continued research. In the meantime, the creep strength data obtained upon spheroidized steels is of sufficient importance to justify tentative conclusions.

TABLE I. HEAT TREATMENT, PHYSICAL PROPERTIES AND RESULT OF CREEP TESTS AT 900 F.

Key[a]	Item	Heat Treatment[b] (All Specimens Drawn 1200 F.—4 hr.—F.C.)	Spheroidized at 1292 F., hr.	Physical Properties Before and After Creep								Creep Stress for Various Rates per 100,000 hr., psi.			Figure
				Time	Tensile Strength, psi.	Elastic Limit, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Rockwell Hardness, "B" Scale	Charpy Impact, Keyhole Notch, ft-lb.		10%	1%	0.1%	
										Room Temperature	900 F.				
FNI	909		Draw only	Before	68 100	39 000	35.5	64.4		36.9					2
				After	70 600	46 800	36.4	64.1	75.5	27.7	28.5	27 000	20 000	1 500	14(*a*)
FNS	910	1560 F.—8 hr.—A.C.	55	Before	63 200	37 000	36.0	67.0		25.6					3
				After	63 500	35 200	35.0	60.5	70.5	18.2	12.2	12 700	9 200	6 600	14(*a*)
FNM	962		1500	Before	57 700	26 000	36.5	59.4	55.0	9.0					4
				After	56 100	30 200	37.2	60.0		2.1	19.5	10 000	8 100	6 400	14(*a*)
FAI	905		Draw only	Before	67 100	35 000	34.5	58.0		26.3					5
				After	65 400	37 400	37.1	59.0	72.0	19.5	20.1	16 000	14 100	12 500	14(*b*)
FAS	906	1560 F.—8 hr.—F.C.	180	Before	63 700	35 000	36.0	63.0		20.8					6
				After	60 600	36 500	38.4	62.9	69.0	5.1	20.1	12 700	10 200	8 000	14(*b*)
FAM	961		1500	Before	60 200	26 000	33.5	62.2	60.1	8.5					7
				After	59 600	29 500	37.2	71.4		5.1	11.1	10 600	8 800	7 000	14(*b*)
CNI	911		Draw only	Before	70 900	41 000	31.5	63.2		24.0					8
				After	79 000	31 400	32.9	56.7	84.5	18.8	11.1	30 000	24 300	19 700	14(*c*)
CNS	912	1750 F.—8 hr.—A.C.	55	Before	67 200	43 000	33.0	68.4		37.8					9
				After	63 400	37 400	39.3	68.9	72.0	20.8	18.2	21 000	16 500	13 000	14(*c*)
CNM	964		1500	Before	56 900	23 000	36.0	58.9	59.5	2.6					10
				After	58 100	31 200	40.7	58.3			19.5	8 800	7 200	5 900	14(*c*)
CAI	907		Draw only	Before	63 600	32 000	34.0	56.9		22.8					11
				After	65 500	29 100	37.8	55.6	72.0	16.3	18.2	16 600	13 800	11 600	14(*d*)
CAS	908	1750 F.—8 hr.—F.C.	180	Before	61 900	26 000	35.0	60.8		2.5–3.8					12
				After	62 400	28 100	34.9	60.4	68.0	2.1	9.0	14 600	10 000	7 250	14(*d*)
CAM	963		1500	Before	60 400	26 000	35.0	59.4	64.0	2.6					13
				After	57 100	30 200	36.4	61.7		2.6	19.5	12 000	8 400	6 400	14(*d*)

[a] *F* = Fine Grain Size. *C* = Coarse Grain Size. *N* = Normalized. *A* = Annealed. *I* = Initial, Not Aged. *S* = Spheroidized. *M* = Massed Cementite.

[b] Heat Treatment: F.C. = Furnace Cooled. A.C. = Air Cooled.

PLAN OF TESTS

A stock of carbon-molybdenum steel from one heat was first divided according to heat treatments into four groups. Each group is composed of fine grain (*F*) and coarse grain (*C*) steel, normalized (*N*) and annealed (*A*) as shown in Fig. 1. The four groups of the steel were given preliminary tests for time to spheroidize at temperature without stress. Each group was then further subdivided into three conditions of carbide spheroidization as shown in Fig. 1. Two sets of steel thus subdivided into twelve different conditions were then tested in long-time creep, one set at 900 F. and the other at 1000 F.

MATERIAL

The material used in this investigation is a weldable ½ per cent molybdenum steel (heat 57,328) with the chemical composition 0.17 per cent carbon, 0.42 per cent molybdenum, 0.88 per cent manganese, 0.20 per cent silicon, 0.016 per cent aluminum, and 0.007 per cent Al_2O_3. It is from a commercial heat of open-hearth, aluminum-killed steel rolled into a 4 in. thick plate. After preliminary heat treatment and spheroidizing tests, eight bars $2\frac{1}{4}$ by 4 by 36 in. were machined from the plate. These eight bars were divided into four groups of two bars each. These four groups were heat treated as shown in Fig. 1 and Table I to the base conditions *FNI*, *FAI*, *CNI* and *CAI*. Each bar was sawed into six bars $\frac{7}{8}$ by 1 by 36 in. making twelve bars per base group. In each group four bars were retained in the initial condition *I*; four bars were spheroidized to condition *S*; and four bars to condition *M*. From this stock specimens were prepared for creep and other tests.

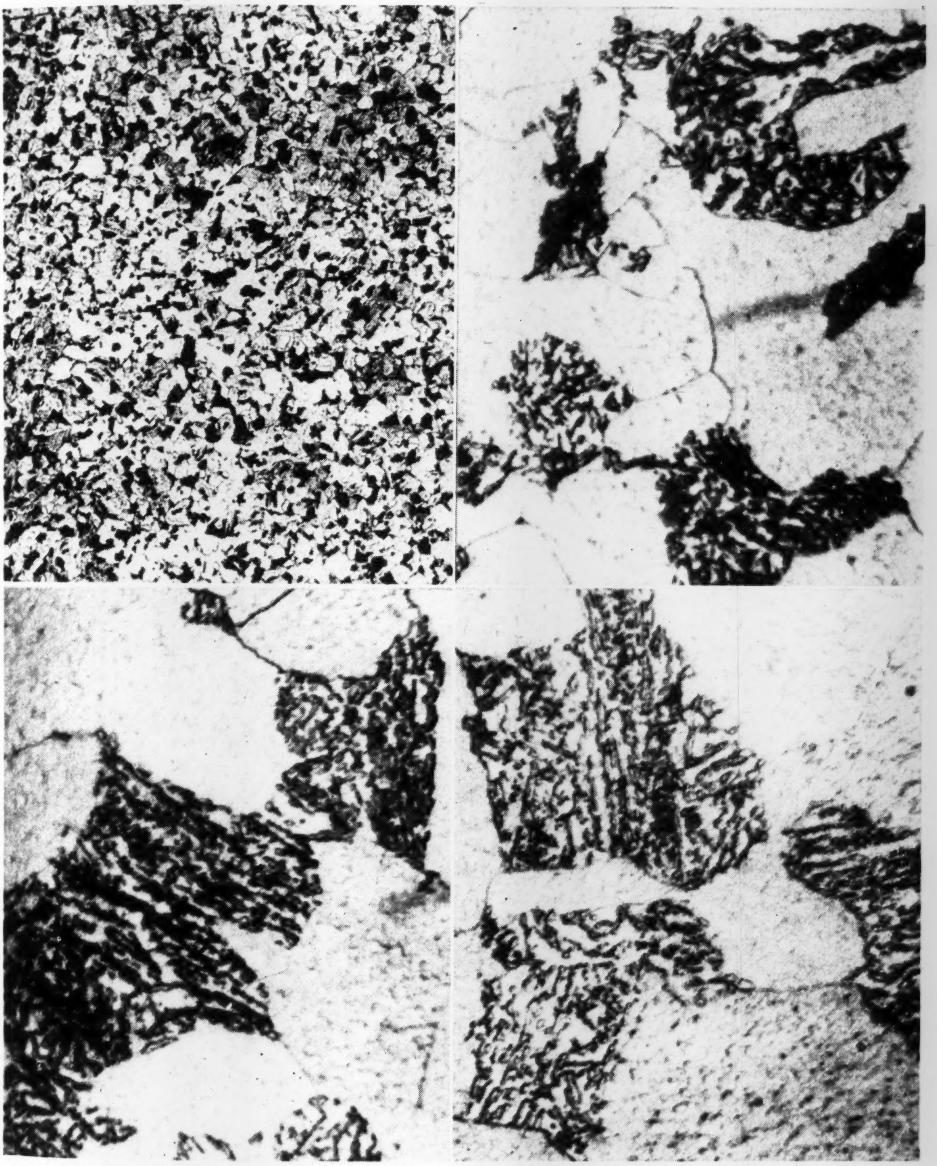
TABLE II. HEAT TREATMENT, PHYSICAL PROPERTIES AND RESULT OF CREEP TESTS AT 1000 F.

Key[a]	Item	Heat Treatment[b] (All Specimens Drawn 1200 F.—4 hr.—F.C.)	Spheroidized at 1292 F., hr.	Physical Properties Before and After Creep						Charpy Impact, Keyhole Notch, ft-lb.		Creep Stress for Various Rates per 100,000 hr., psi.			Figure
				Time	Tensile Strength, psi.	Elastic Limit, psi.	Elongation, in 2 in., per cent	Reduction of Area, per cent	Rockwell Hardness "B" Scale	Room Temperature	1000 F.	10%	1%	0.1%	
FNI	897		Draw only	Before	68 100	39 000	35.5	64.4		36.9					2
				After	69 500	45 600	30.0	60.8	74.5	29.1	15.1	20 000	7 200	2 500	14(*a*)
FNS	898	1560 F.—8 hr.—A.C.	55	Before	63 200	37 000	36.0	67.0		25.6					3
				After	63 500	33 200	35.7	64.6	69.5	22.8	30.8	6 000	3 600	1 850	14(*a*)
FNM	966		1500	Before	57 700	26 000	36.5	59.4	55.0	9.0					4
				After	56 100	32 300	39.3	60.0	61.0	3.4	18.8	4 500	2 700	1 780	14(*a*)
FAI	901		Draw only	Before	67 100	35 000	34.5	58.0		26.3					5
				After	67 000	36 400	35.0	60.9	60.9	19.5	28.5	17 500	7 600	2 900	14(*b*)
FAS	902	1560 F.—8 hr.—F.C.	180	Before	63 700	35 000	36.0	63.0		29.8					6
				After	59 500	35 300	37.8	65.5	66.5	7.0	25.6	6 300	3 400	1 850	14(*b*)
FAM	965		1500	Before	60 200	26 000	33.5	62.2	60.0	8.5					7
				After	59 200	31 200	40.0	61.5	64.0	5.1	26.5	4 700	3 100	2 050	14(*b*)
CNI	899		Draw only	Before	70 900	41 000	31.5	63.2		24.0					8
				After	67 600	35 300	38.5	61.6	76.5	24.0	30.8	22 000	11 000	3 800	14(*c*)
CNS	900	1750 F.—8 hr.—A.C.	55	Before	67 200	43 000	33.0	68.4		37.8					9
				After	58 600	39 500	37.9	67.0	76.0	10.0	31.7	9 600	6 400	2 450	14(*c*)
CNM	968		1500	Before	56 900	23 000	36.0	58.9	59.5	2.6					10
				After	56 600	31 200	37.8	61.5	60.0	1.8	18.8	4 500	2 500	1 100	14(*c*)
CAI	903		Draw only	Before	63 600	32 000	34.0	56.9		22.8					11
				After	66 600	27 000	32.5	55.6	72.5	14.5	18.2	14 200	10 800	3 500	14(*d*)
CAS	904	1750 F.—8 hr.—F.C.	180	Before	61 900	26 000	35.0	60.8		2.5–3.8					12
				After	62 400	27 000	34.0	61.6	67.0	2.6	35.3	6 400	4 350	2 100	14(*d*)
CAM	967		1500	Before	60 400	26 000	35.0	59.4	64.0	2.6					13
				After	60 600	31 200	37.1	59.8	63.5	2.6	19.5	4 900	2 900	1 650	14(*d*)

[a] *F* = Fine Grain Size. *C* = Coarse Grain Size. *N* = Normalized. *A* = Annealed. *I* = Initial, Not Aged. *S* = Spheroidized. *M* = Massed Cementite.

[b] Heat Treatment: F.C. = Furnace Cooled. A.C. = Air Cooled.

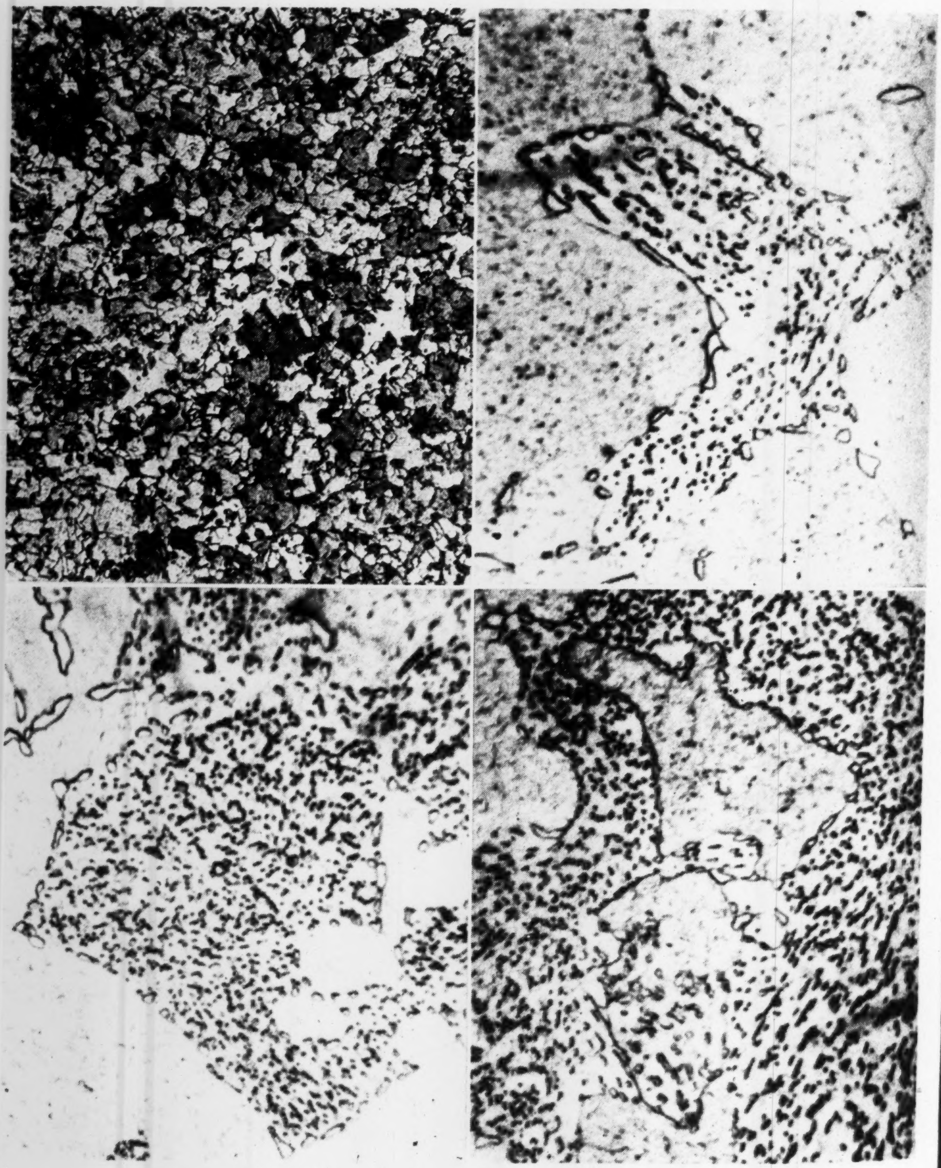
Before creep test (×100). Before creep test (×1500).



After creep at 900 F. (×1500). After creep at 1000 F. (×1500).

FIG. 2.—Microstructure of Fine Grained, Normalized Carbon-Molybdenum Steel in Initial Tempered Condition.

FNI in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 20,000 and 7200 psi. at 900 and 1000 F., respectively.

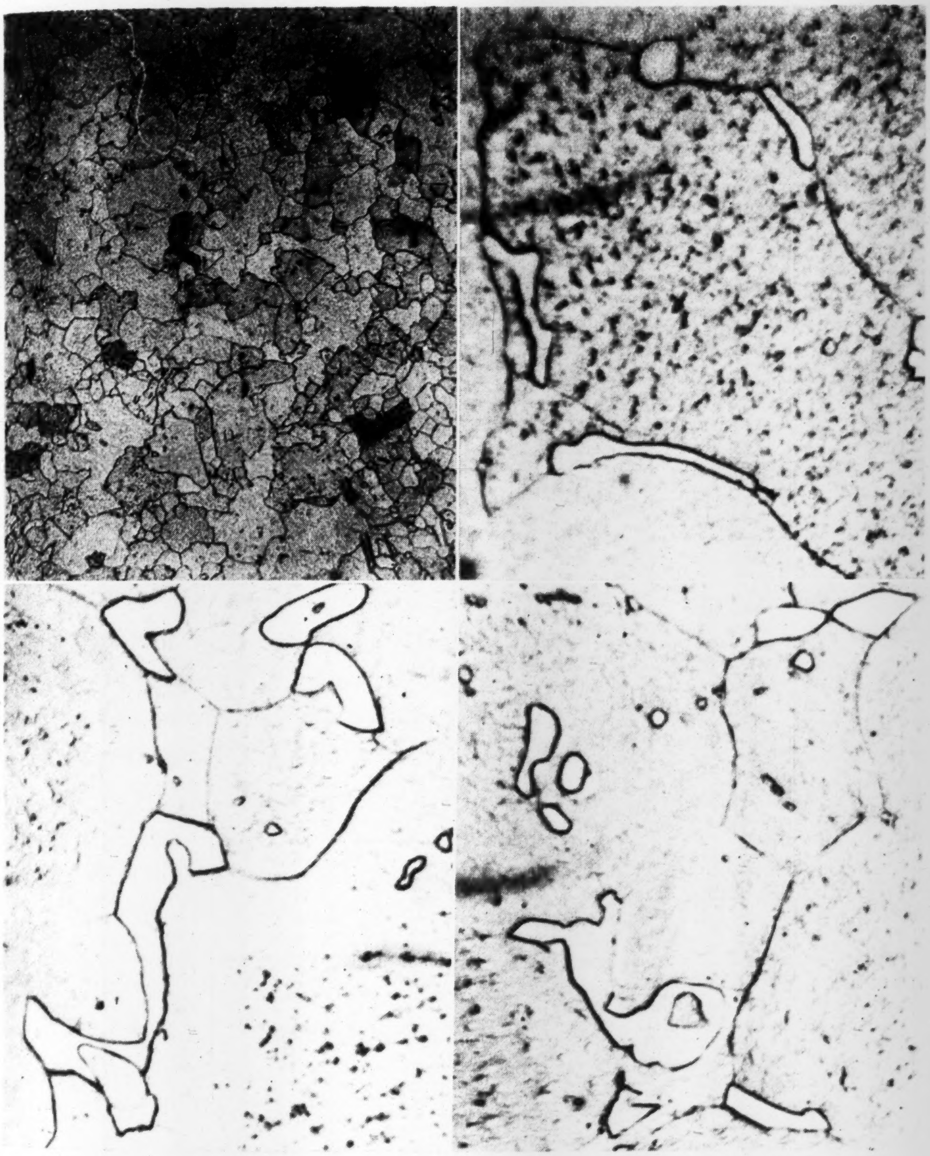


Before creep test (×100). Before creep test (×1500).

After creep at 900 F. (×1500). After creep at 1000 F. (×1500).

FIG. 3.—Like Fig. 2 Except Spheroidized 55 hr. at 1292 F.

FNS in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 9200 and 3600 psi. at 900 and 1000 F., respectively.

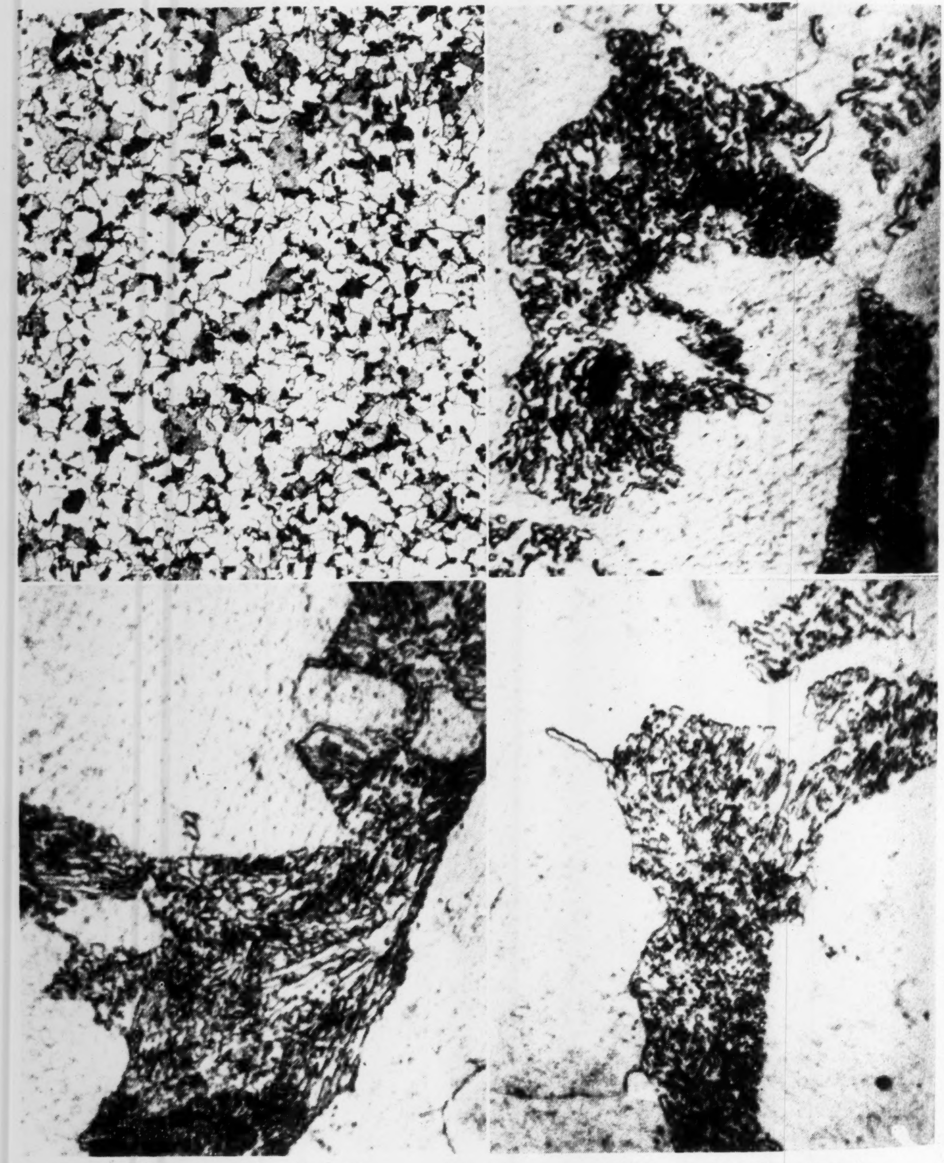


Before creep test (×100). Before creep test (×1500).

After creep at 900 F. (×1500). After creep at 1000 F. (×1500).

FIG. 4.—Like Fig. 2 Except Spheroidized 1500 hr. at 1292 F.

FNM in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 8100 and 2700 psi. at 900 and 1000 F., respectively.



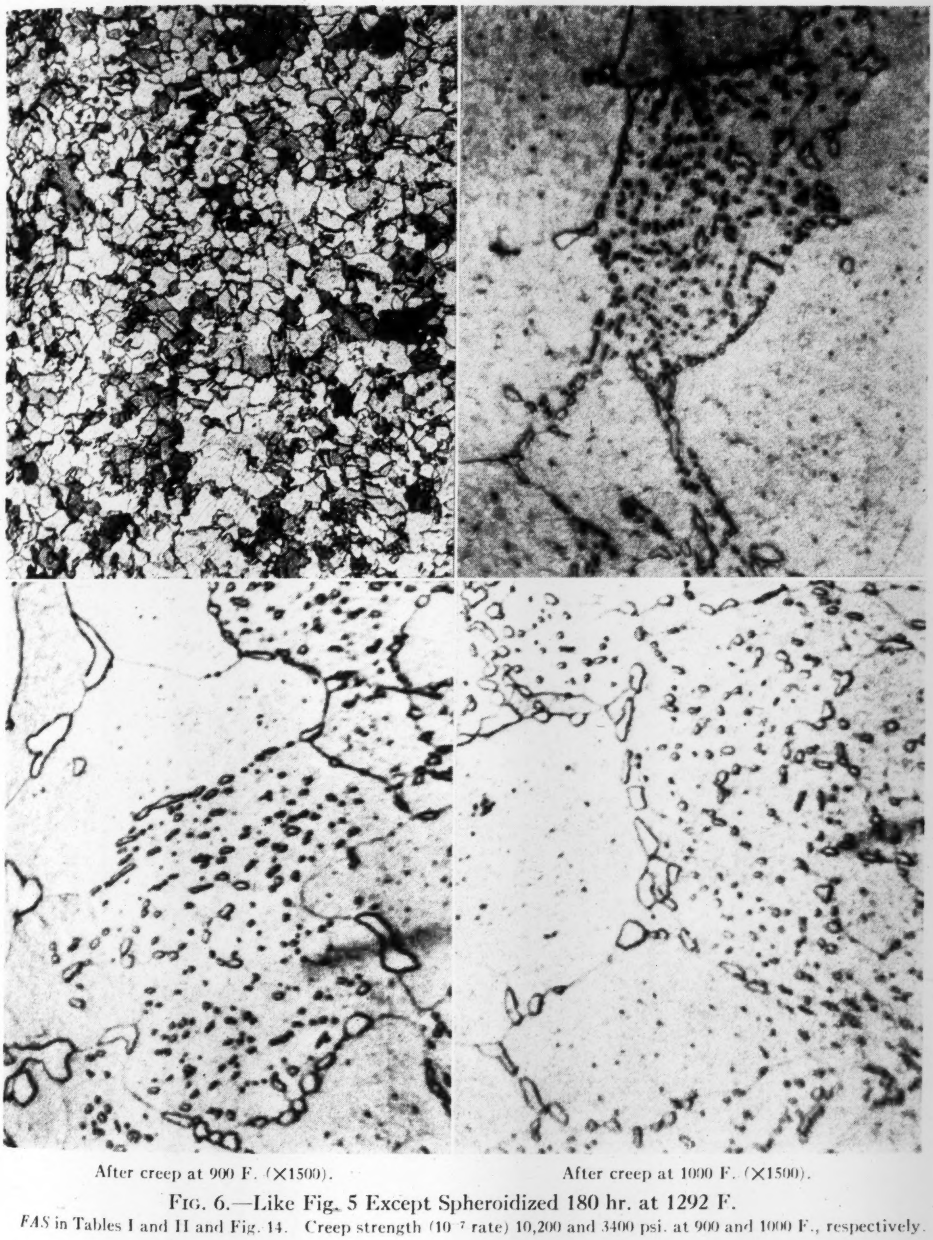
Before creep test (×100).

Before creep test (×1500).

After creep at 900 F. (×1500).

After creep at 1000 F. (×1500).

FIG. 5.—Microstructure of Fine Grained, Annealed Carbon-Molybdenum Steel in Initial Condition. *FA1* in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 14,100 and 7600 psi. at 900 and 1000 F., respectively.

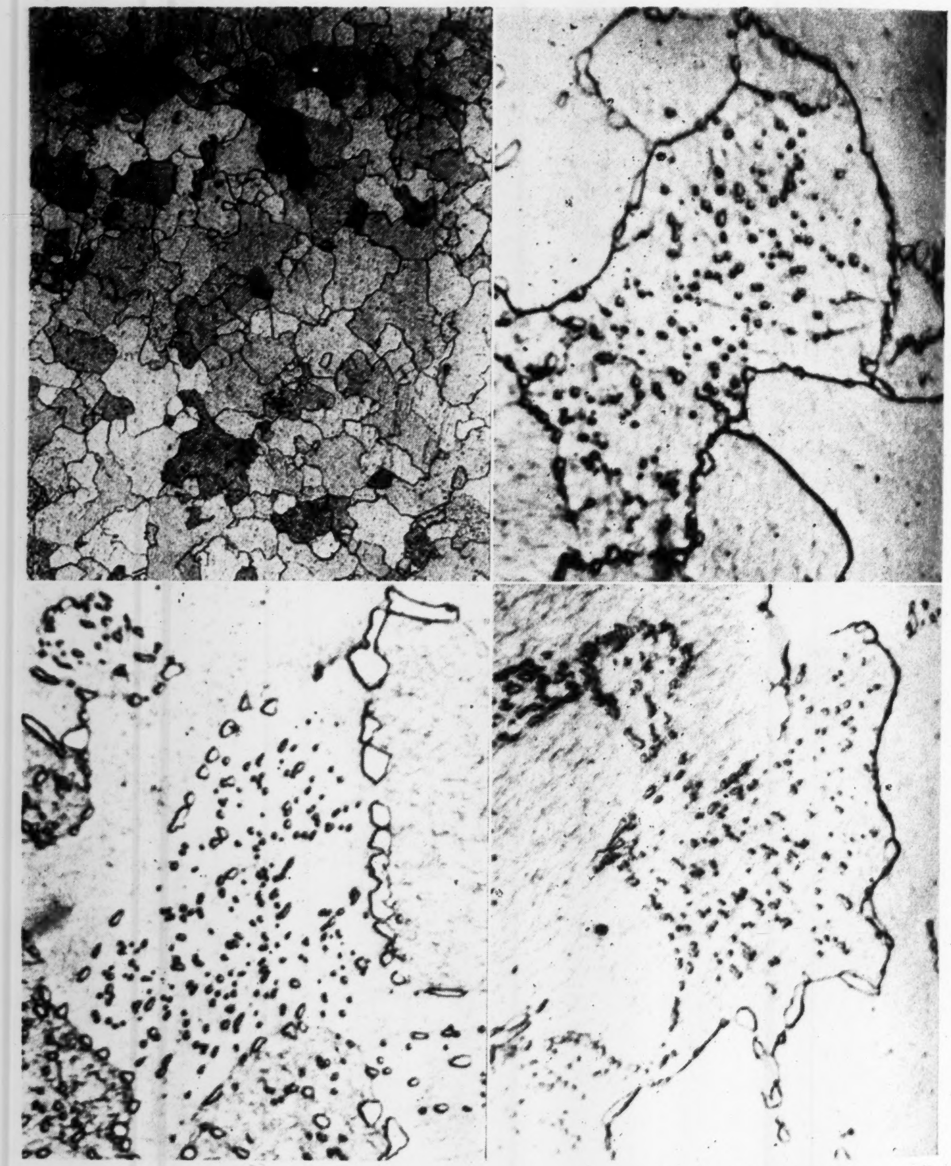


Before creep test (×100). Before creep test (×1500).

After creep at 900 F. (×1500). After creep at 1000 F. (×1500).

FIG. 6.—Like Fig. 5 Except Spheroidized 180 hr. at 1292 F.

FAS in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 10,200 and 3400 psi. at 900 and 1000 F., respectively.

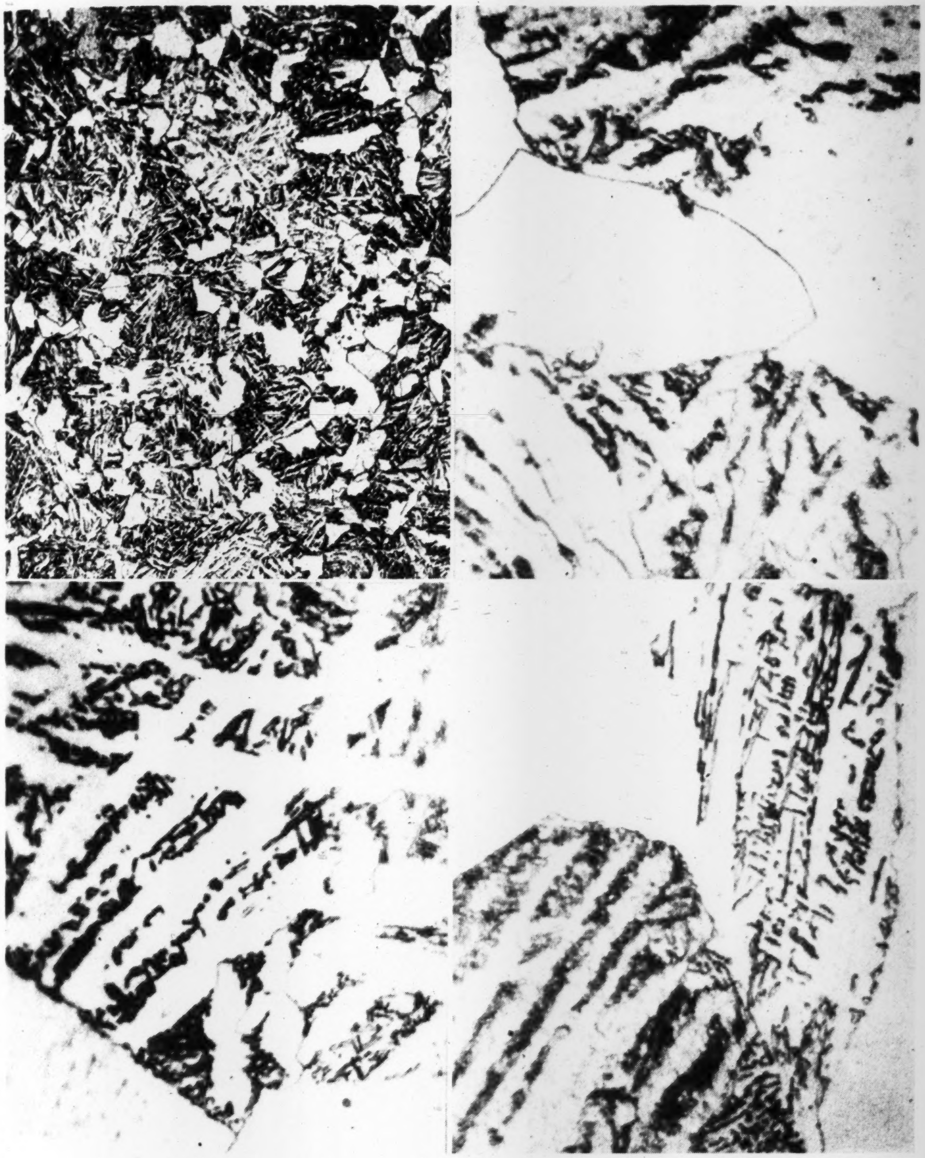


Before creep test (×100). Before creep test (×1500).

After creep at 900 F. (×1500). After creep at 1000 F. (×1500).

FIG. 7.—Like Fig. 5 Except Spheroidized 1500 hr. at 1292 F.

FAM in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 8800 and 3100 psi. at 900 and 1000 F., respectively.



Before creep test (×100).

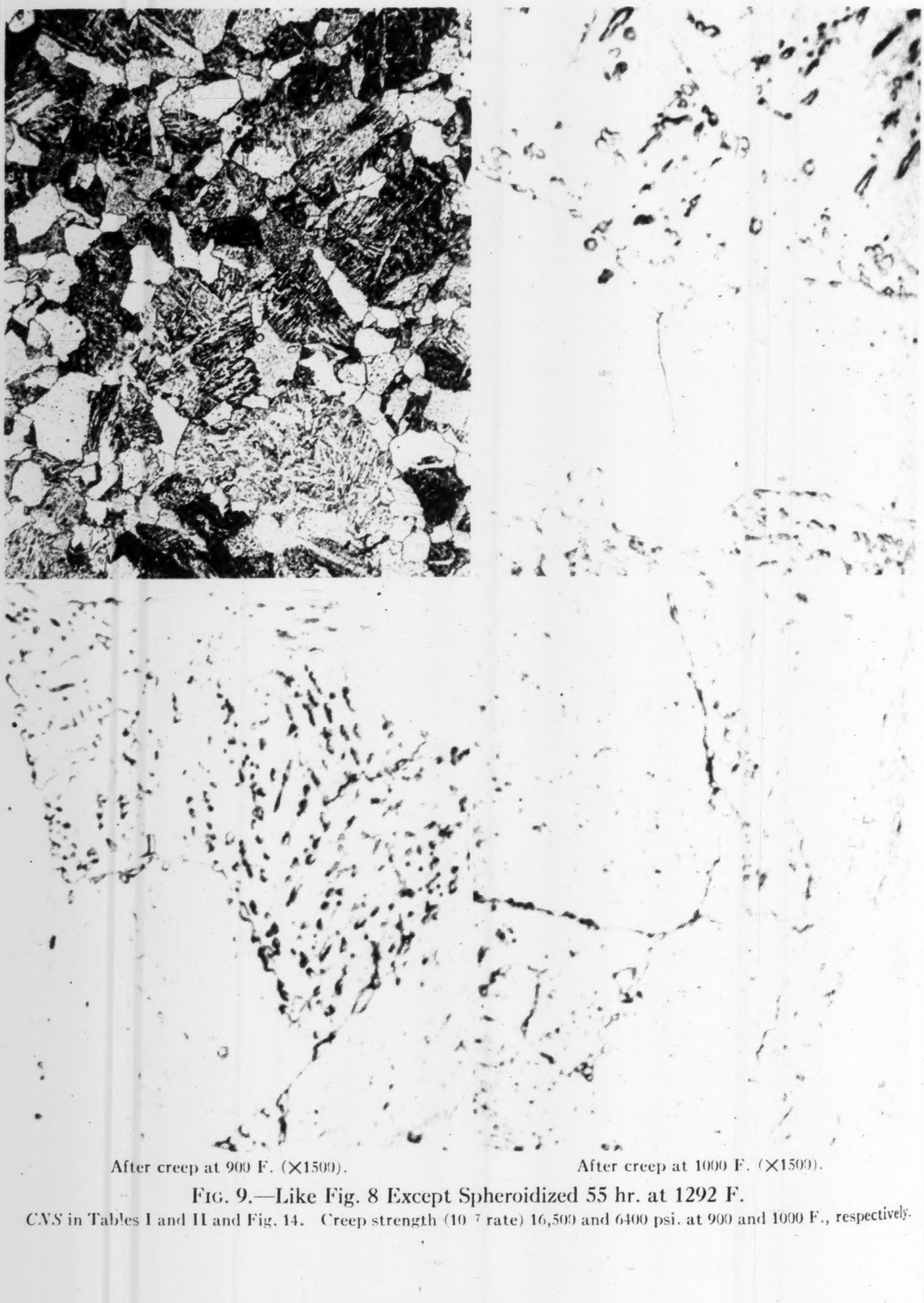
Before creep test (×1500).

After creep at 900 F. (×1500).

After creep at 1000 F. (×1500).

FIG. 8.—Microstructure of Coarse Grained, Normalized Carbon-Molybdenum Steel in Initial Tempered Condition.

CNI in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 24,300 and 11,000 psi. at 900 and 1000 F., respectively.

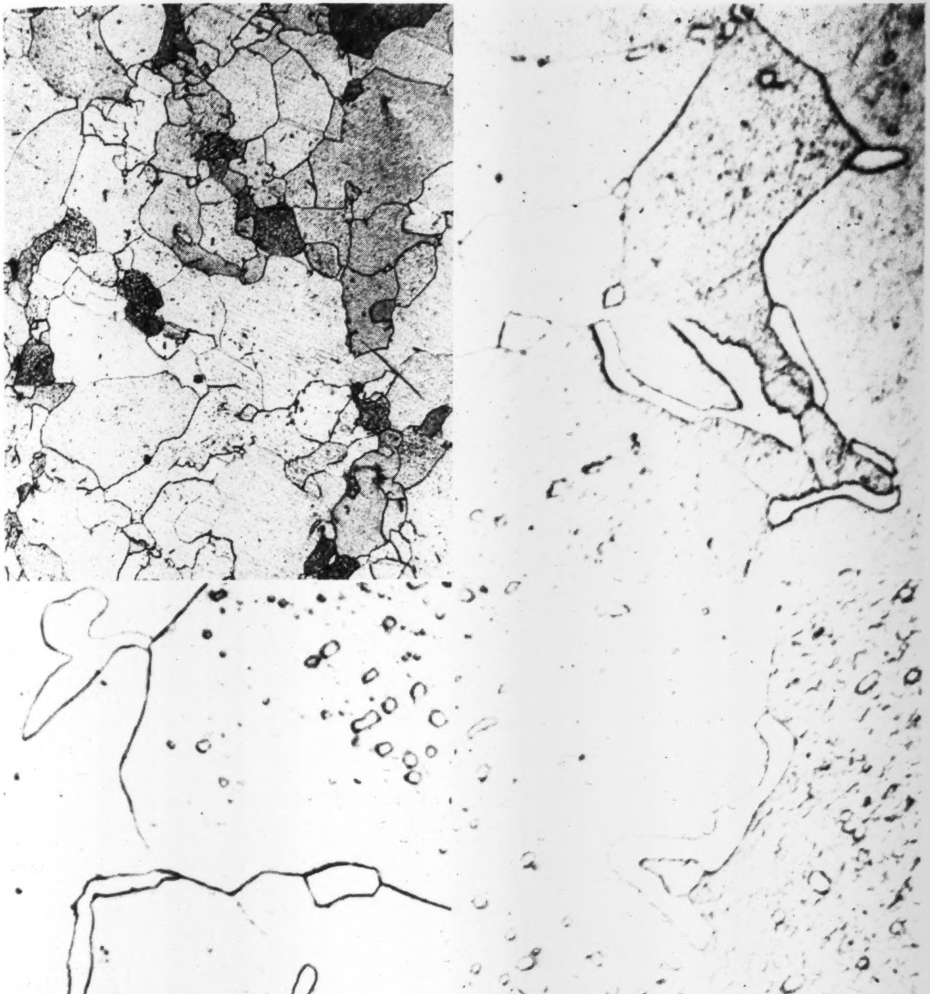


Before creep test (×100). Before creep test (×1500).

After creep at 900 F. (×1500). After creep at 1000 F. (×1500).

FIG. 9.—Like Fig. 8 Except Spheroidized 55 hr. at 1292 F.

CNS in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 16,500 and 6400 psi. at 900 and 1000 F., respectively.



Before creep test (×100).

Before creep test (×1500).

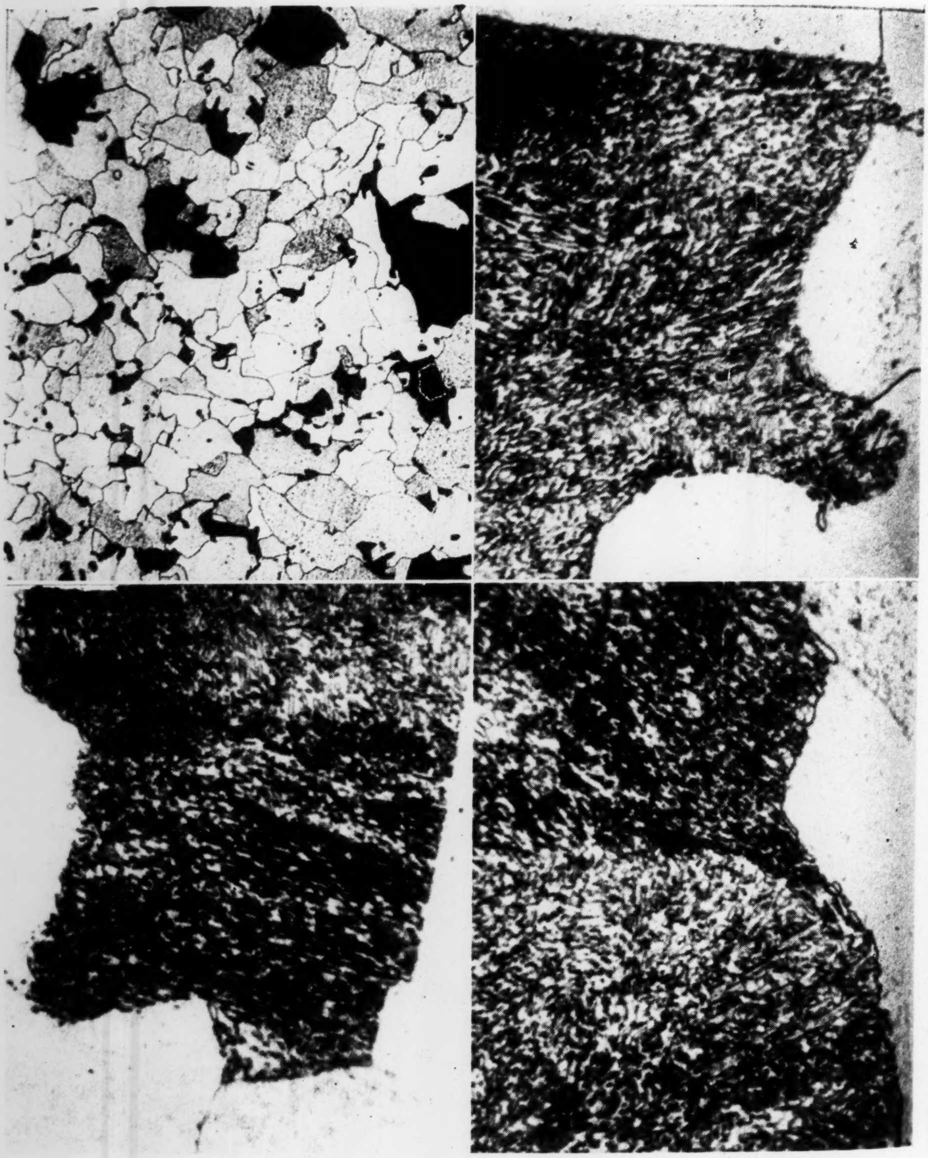
After creep at 900 F. (×1500).

After creep at 1000 F. (×1500).

FIG. 10.—Like Fig. 8 Except Spheroidized 1500 hr. at 1292 F.

CNM in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 7200 and 2500 psi. at 900 and 1000 F., respectively.

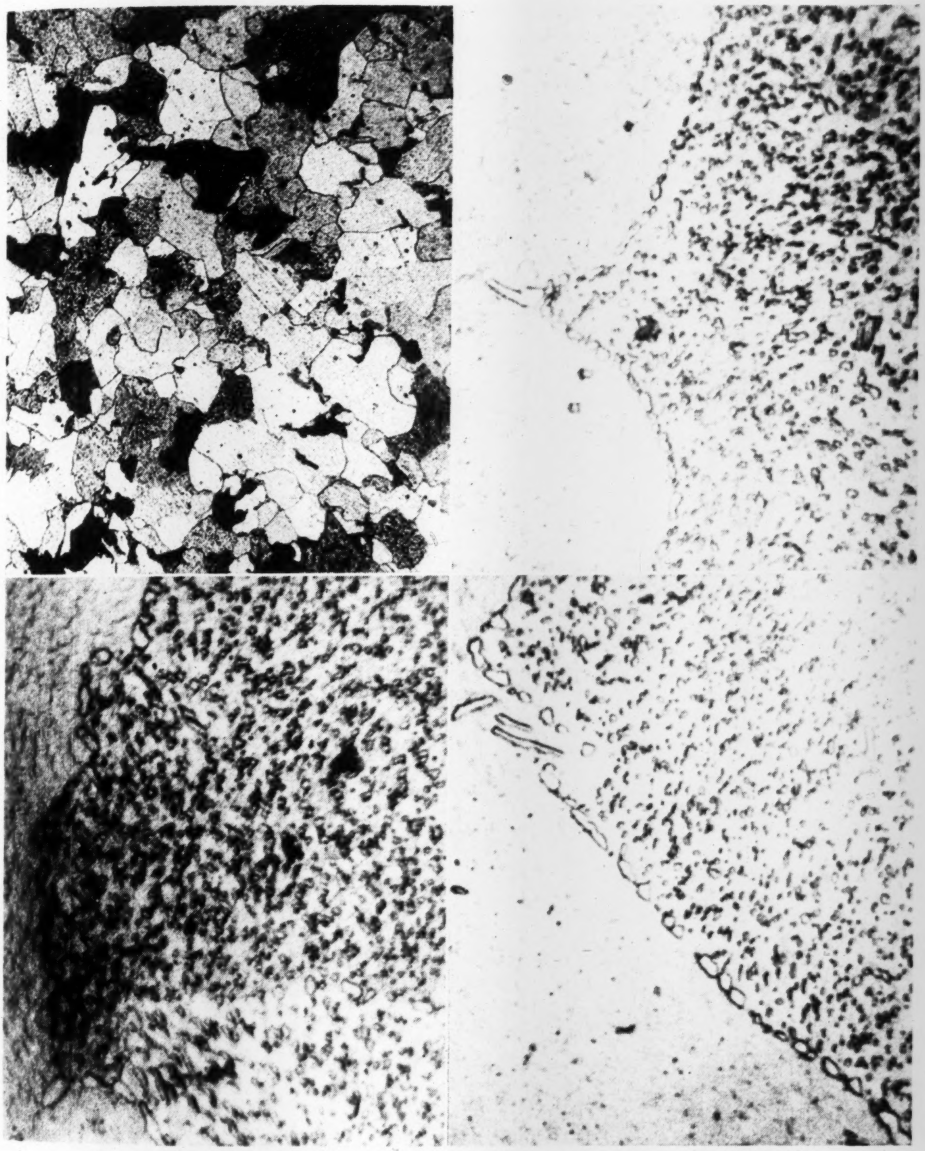
Before creep test (×100). Before creep test (×1500).



After creep at 900 F. (×1500). After creep at 1000 F. (×1500).

FIG. 11.—Microstructure of Coarse Grained, Annealed Carbon-Molybdenum Steel in Initial Condition.

CA1 in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 13,800 and 10,800 psi. at 900 and 1000 F., respectively.



Before creep test (×100).

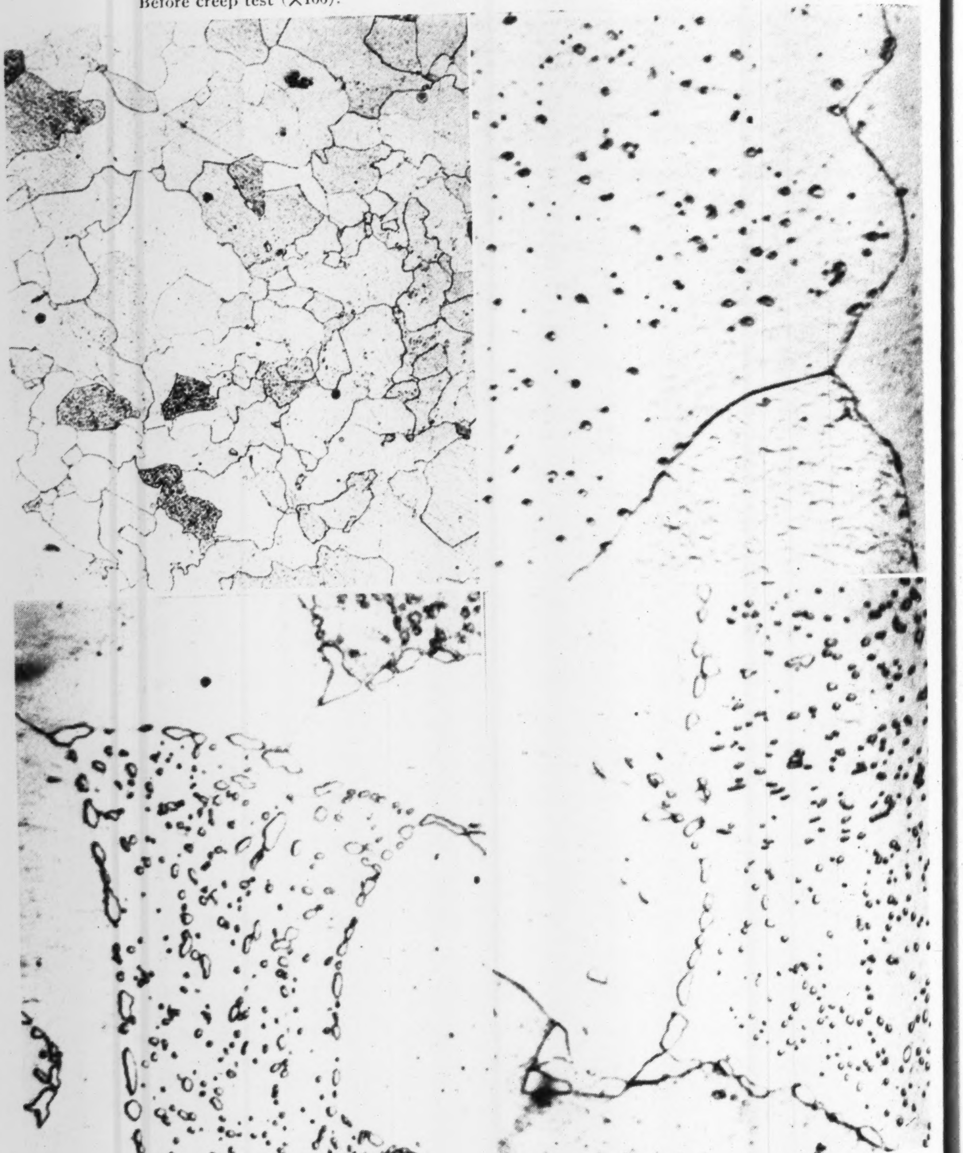
Before creep test (×1500).

After creep at 900 F. (×1500).

After creep at 1000 F. (×1500).

FIG. 12.—Like Fig. 11 Except Spheroidized 180 hr. at 1292 F.

CAS in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 10,000 and 4350 psi. at 900 and 1000 F., respectively.



Before creep test (×100).

Before creep test (×1500).

After creep at 900 F. (×1500).

After creep at 1000 F. (×1500).

FIG. 13.—Like Fig. 11 Except Spheroidized 1500 hr. at 1292 F.

CAM in Tables I and II and Fig. 14. Creep strength (10^{-7} rate) 8400 and 2900 psi. at 900 and 1000 F., respectivel

Preliminary heat treatments for 8 hr. at 1560 F. gave a No. 7 to No. 8 uniform grain size, treatment at 1650 and 1700 F. resulted in a duplex or mixed grain size, while at 1740 F. annealing gave a No. 4 and normalizing a No. 2 uniform grain size as shown in Figs. 2, 5, 8, and 11, respectively. This steel has a narrow grain coarsening temperature range that is caused by a grain growth inhibitor introduced during the deoxidization process. A heat treatment within this grain coarsening temperature range results in a mixed grain size which is objectionable in steels for high-temperature service; and the mixed grains, once established, tend to persist through subsequent heat treatments. The grain referred to is that which actually exists in the steel structure during the test and the service life of the material. It is termed "structural grain size" in A.S.T.M. Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for Service at Temperatures from 750 to 1000 F. (A 206 – 40T)[2] and "refers to the predominant grain as evidenced by ferrite grains, Widmanstaetten areas, or pearlite patches," using the A.S.T.M. Tentative Classification of Austenite Grain Size in Steels (E 19 – 39 T),[3] as the unit of measurement.

CARBIDE SPHEROIDIZATION

The spheroidization of carbides in relation to time and service life presented a problem. Some investigators have used an arbitrarily chosen photomicrograph as a standard for the degree of spheroidization; so that when a new photomicrograph is taken as standard the data are not comparable. A solution of the problem was suggested in an unpublished thesis submitted to the University of Michigan by J. H. Taylor, on "A Study of Spheroidization in Steels at Temperatures Below the Critical Range," dated August, 1934. This report defines a definite end standard of measurement for full spheroidization which can be duplicated in tests by other laboratories.

Condition *I* in Fig. 1 is the initial condition of the steel without aging or spheroidizing treatment. Condition *S* is with the cementite fully spheroidized. It is obtained first from the annealed steel as *FAI* (×1500) (Fig. 5) holding at temperature 1292 F. without stress, and examining the microstructure at different periods of time. The number of hours at which the last trace of lamellae formation is no longer discernable is considered the end standard for full spheroidization (*FAS* (×1500) in Fig. 6). Similarly, the same steel in the normalized condition as *FNI* (× 1500) (Fig. 2) is said to be fully spheroidized when the spheroids as *FNS* (×1500) (Fig. 3) are of the same size as those obtained in the annealed standard described above. The same process was repeated for the coarse grained steels. Taylor proved on different carbon steels with various annealing temperatures that this standard is independent of lamellar spacing. Taylor's time ratio of annealed to normalized conditions, 80 to 25 for 0.13 carbon steel, is practically the same as that found here, 180 to 55 for the carbon-molybdenum steel.

For condition *M* with the massed spheroids there was no previous guide so it was proposed to continue the spheroidizing time until the spheroids were fully coalesced leaving the ferrite matrix clear. This condition was obtained only for *FNM* steel as shown in Fig. 4 at 1500 hr. and 1292 F. At the magnification used in these photomicrographs the spheroids and masses of cementite have definite boundaries while the more stable intermetallic compound, shown by E. R. Parker[4] to be Fe_3Mo_2, precipitates slowly into fine particles without sharp boundaries. Figures 7, 10 and 13 still contain large spheroids within the matrix of the grains. It was assumed that the 1500 hr. would be equivalent to a reasonably long service life and was therefore used in the tests.

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[2] 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 252.

[3] 1939 Book of A.S.T.M. Standards, Part I, p. 1175.

SERVICE LIFE

The relationship between spheroidizing time and operating temperature of the apparatus is necessary to estimate the service life of the steel. Data are not sufficient to formulate a time-temperature relationship for the massed spheroids M. The fully spheroidized condition S follows the Langmuir-Dushman exponential equation for diffusion in solids,

$$t = Ae^{\frac{b}{T}}$$

where t = time,
T = the absolute temperature,
e = the Naperian base,
A = the test constant for composition and condition of the steel, and
b = a constant for pearlitic steels.

The average value of b calculated from thirteen tests made by Taylor was 45,000; the largest value being 46,260 and the smallest 44,280, when T is in degrees Absolute Fahrenheit. Seven of the tests were made on differently alloyed steels up to 5 per cent chromium, three tests were made on each of low, medium, and high-carbon steels, and three tests covering different conditions were made on the same low-carbon steel. Graphs of the reciprocal of absolute temperature against logarithm of time showed a straight line both for the formula and for the spheroidizing tests. Tests ranged from 1300 F. to 1100 F. and an extrapolation of the straight line to 1000 F. and 900 F. creep temperatures is considered permissible from the standpoint of service operating hours.

It is the difference between testing and operating conditions which is the unknown quantity. The presence of a working stress should hasten atomic movements, increase the rate of spheroidization and influence recrystallization. Such tests are in progress, on tapered test specimens under stress, and will require several years for completion. Two tests made by Taylor on carbon steel under stress, indicate a possible reduction to two thirds of the time that is required for full spheroidization without stress. This leads to an approximation that, for the same temperature, 100,000 hr. of spheroidizing time is equivalent to eight instead of eleven years of service time.

CREEP TESTS

Creep tests were made on each of the twelve conditions in Fig. 1 at 900 F. and upon duplicate bars at 1000 F. The tests were made by the step down stress method with the total elastic plus plastic extension limited to 0.002 in. per inch. During the last stage of the test, a constant stress is held for 500 to 1000 hr. to obtain a definite creep rate below 1 per cent per 100,000 hr. It is not contended that such rates will remain unchanged for 100,000 hr.; they are the specific rates of flow obtained during the creep test. The duration of tests ranged from 2150 to 3700 hr.

Creep test results are summarized in Table I for 900 F. and in Table II for 1000 F. Key letters from Fig. 1 indicate the condition of the steel in each test. The tables list the heat treatments, physical properties and creep stresses for each condition of the steel and refer to the figure numbers containing the microstructures and creep test plots.

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[4] E. R. Parker, "The Development of Alloys for Use at High Temperatures Above 1000 F.," *Transactions*, Am. Soc. Metals., Vol. XXXVIII, No. 4, December, 1940, pp. 797-810.

Figures 2 to 13 consist of four photomicrographs for each of the twelve conditions of steel. Two of the photomicrographs were taken before creep, one at 100 magnification to show the structural grain size, and the other at 1500 magnifications to show the carbide formations. The other two photomicrographs were made after creep test, one at 900 F. and the other at 1000 F. Both were taken at 1500 magnifications for comparison with the carbide conditions.

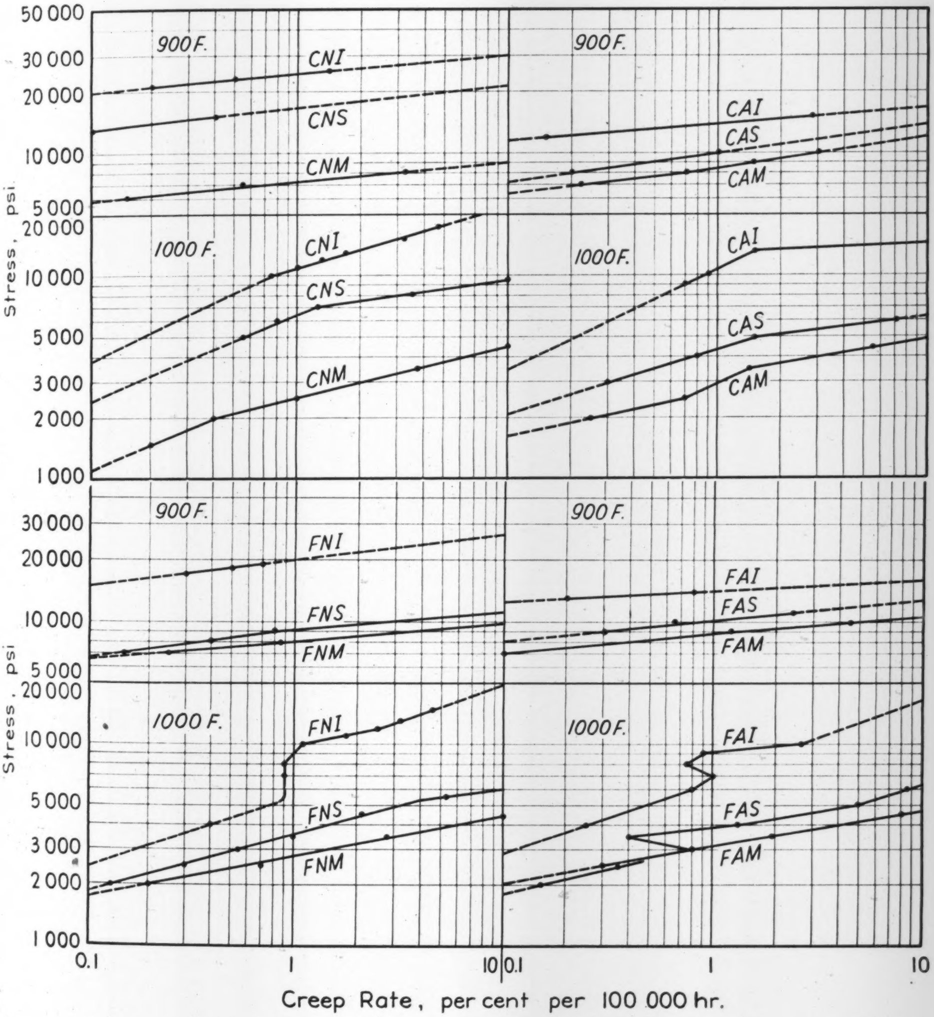

FIG. 14.—Creep Stress and Rate of Creep for 0.5 per cent Molybdenum Steel with Differently Spheroidized Microstructures.

Figure 14 shows the relationship between stress and creep rate for each of the twelve conditions at 900 F. and 1000 F. Of the tests at 1000 F., all curves except two contain a change in the slope. The two exceptions are the low-creep-strength, fine-grained steels with the carbides coalesced into large masses, *FNM* and *FAM*, which show straight lines. Several other fine-grained steels gave very irregular curves. In particular, *FNI*, *FAI*, and *FAS* suffered a very appreciable drop in creep strength at about the time the load was reduced to approximately that which caused a rate of 1 per cent per 100,000 hr. These bars indicate a higher level of creep strength at the start than at the conclusion of the test, the transition occurring about halfway through the test. Similar behavior has been noted in previous tests at this higher creep temperature and, in view of the care exercised, the data are scarcely open to question.

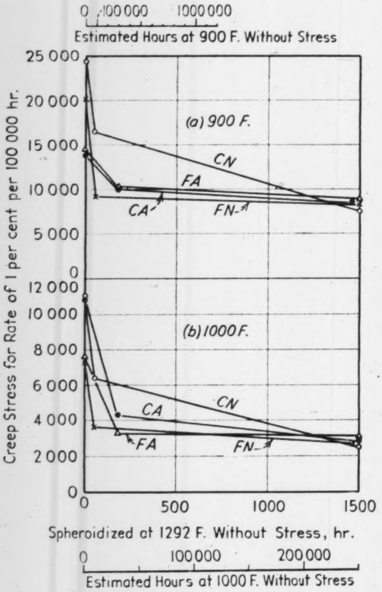

FIG. 15.—Creep Stress at 900 and 1000 F. for 0.5 per cent Molybdenum Steel when Spheroidized for Different Amounts of Time.

There are also two tests which lack low enough rates to give reliable indications as to the creep stress for a rate of 0.1 per cent per 100,000 hr. Thus *FNI* at 1000 F. has been extrapolated as shown by the dashed line with the same slope as *FAI* at 1000 F. Similarly *CNI* at 1000 F. is extrapolated with the same slope as *CAI* at 1000 F.

TEST RESULTS

In Fig. 15 creep stress is plotted against the time required to spheroidize the carbides in the steel. The linear scale gives a perspective to the time required by the temperature to produce changes in the steel. Scales have been added for the equivalent time at the creep temperature without stress at 900 and 1000 F., using the spheroidizing formula previously discussed under the heading "service life."

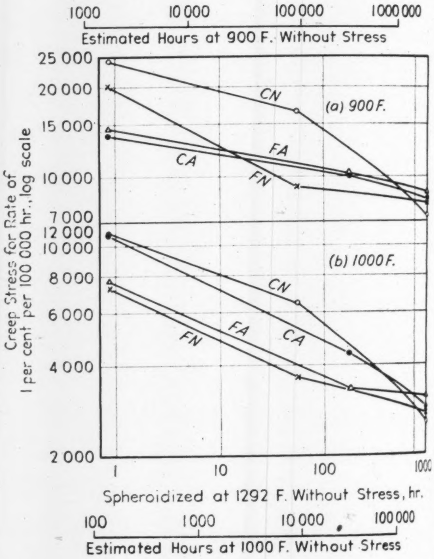

FIG. 16.—Log Plot of Creep Stress at 900 and 1000 F. *versus* Spheroidizing Time Without Stress for 0.5 per cent Molybdenum Steel.

Considering that a possible effect of the presence of stress is to reduce the spheroidizing time to two thirds, an approximation for service time at the creep temperatures can be made by considering that the test points at 55, 180, and 1500-hr. spheroidizing time at 1292 F. are equivalent to 7, 23, and, 200 yr. in Fig.

15 (*a*) and 0.7, 2.3, and 20 yr. in Fig. 15 (*b*), of service time at the temperature of the creep test. During these service periods the creep stress for a constant value in the creep rate has decreased to between 72 and 46 per cent of the initial creep stress in 23 yr. operation at 900 F. The more rapid decrease in creep strength occurs in the 1000 F. test where from 58 to 40 per cent of the initial stress is obtained at 2.3 yr., and from 41 to 23 per cent in 20 yr. of 1000 F. operation.

Figure 16 shows the same data plotted on a log-log scale to produce more nearly straight lines. The initial creep conditions are placed at 0.9 hr. which is equivalent to the 4-hr. draw at 1200 F. Figure 16 shows that at 900 F. creep temperature, the normalized structure *N* is the major factor in the creep strength; when the normalized structure is combined with the coarse grain there is produced the superior condition of the steel for 900 F. creep strength. At 1000 F. creep temperature, the coarse grain is the predominating factor in creep strength, and whether the steel is normalized or annealed is of minor importance.

The physical properties for the twelve conditions of the same heat of steel are also included in Tables I and II. In the "before" and "after creep" tension tests for the initial condition *I*, all fine-grained steel increased and all coarse-grained steels decreased in elastic limit. There is no trend to the changes in physical properties during creep test for the spheroidized condition *S*. For the massed spheroid condition *M*, however, all creep items had a considerable increase in the proportional limit, elongation and reduction in area between the "before" and "after creep" tension tests. There also occurred, during the spheroidizing time between conditions *S* and *M*, a recrystallization or grain growth of two grain size numbers in all of the condition *M* steels as shown in Figs. 2 to 13.

The Charpy tests with keyhole notched specimens, listed in Tables I and II, are limited in number by the size of creep specimens to single tests only. The keyhole notch type of test does not correlate with the endurance properties of the steel, but is related to the notch sensitivity of the material for brittle failure under blows. This is propitious for long service life at high temperature as there is a large decrease in the foot-pounds as the spheroidization advances. All of the steels in the massed spheroid condition *M* and the annealed, fully spheroidized steels *AS* have seven or less foot-pounds at room temperature. All of the initial conditions *I* and the normalized, fully spheroidized steels *NS* have higher values. In all conditions of the steel, the notched-bar strengths are high at the elevated temperatures.

SUMMARY

While the initial creep stresses vary greatly for the different conditions of the steel, in approximately 20 yr. of operation the creep stress for some items decreases to 46 per cent at 900 F. and to 23 per cent at 1000 F.

In selection of treatment for this carbon-molybdenum steel, the creep tests indicate that at 900 F. the normalized structure is the major factor in the creep strength; when combined with the coarse grain it produces the strongest steel. At 1000 F. the coarse grain predominates in the creep strength, and normalized or annealed structures are of minor importance.

DISCUSSION

Mr. A. E. White.[1]—Mr. Weaver, in discussing the effect of carbide spheroidization, makes reference to work done by Mr. Taylor in which it was found that normalized steel broke down more rapidly than annealed steel. By interpolating these data, it was found that it would take about 33,000 hr. to change the structure of the steel in question to spheroidized condition at around 925 F.

It is interesting to note that Mr. Weaver checks earlier findings to the effect that better creep properties are obtained in coarse-grained metal than in fine-grained metal when the metal is at heat above the equicohesive temperature. I wish to compliment Mr. Weaver on the very valuable data which he has made available.

Mr. J. J. Kanter.[2]—I should like to ask Mr. Weaver, whether he could give us, in connection with the information already in the paper, some data as to the rupture elongations which result when testing coarse-grained and fine-grained material? Such additional information would greatly enhance the interpretation and application of the data appearing in the paper.

Mr. S. H. Weaver[3] (*author's closure, by letter*).—The author agrees with Mr. White's estimate that the normalized carbon-molybdenum steel requires approximately 4 yr. at 925 F. to attain the fully spheroidized condition *S*. Furthermore, if the time at 925 F. is extended to 20 yr., an interpolation between the *S* and *M* spheroidized conditions in Fig. 16 reduces the creep stress to approximately 50 per cent of the initial creep stress but is still above the working stress in the A.S.M.E. Power Boiler Code, provided the steel has a reasonably large structural grain. We believe these temperatures and working stresses are safe, as evidenced by the number of large steam plants operating above 900 F.

An implied question in these tests is that, due to the rapid increase in the rate of spheroidization at the higher temperatures, 1000 F. is near a practical design limit for this steel in long service life. It was to obtain data for this limiting temperature condition that the usual carbide spheroidization was extended to the agglomerated state *M*.

In reply to Mr. Kanter, the extra stock of material in the twelve conditions shown in Fig. 1 was placed (1) in long-time rupture tests at 900 and 1000 F. with elongation being measured throughout the tests and (2) in carbide spheroidization tests at 1100 and 1200 F. with tapered specimens under constant load to show the effect of various stresses on the rate of spheroidization. Neither series of tests has progressed far enough for conclusions.

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[1] Director of Department of Engineering Research, and Professor of Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

[2] Research Engineer, Crane Co., Chicago, Ill.

[3] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

THE STRESS-STRAIN CHARACTERISTICS OF COLD-ROLLED AUSTENITIC STAINLESS STEELS IN COMPRESSION AS DETERMINED BY THE CYLINDER TEST METHOD

By Russell Franks[1] and W. O. Binder[1]

Synopsis

This paper deals with a method of determining the stress-strain characteristics in compression of thin sections of cold-rolled stainless steels or of other materials. The method is applicable to sections as thin as 0.01 in. and as thick as 0.06 in., and probably can be applied to thinner or thicker sections if the test specimen is properly designed. The compressive stress-strain characteristics of thin cold-rolled sections of stainless steel are important in view of their application in lightweight high-strength structures such as trains, truck trailers, airplanes, etc.

The method consists of cold-forming strip or sheet into cylinders having the proper slenderness ratio and diameter-to-thickness ratio so that when tested in compression they fail symmetrical to the axis. Properly designed specimens can be loaded beyond the yield strength without failing prematurely due to local instability, and the yield strength as determined by the 0.2 per cent offset method can be obtained. The compression properties of material both longitudinal and transverse to the direction of rolling can be determined depending upon the manner in which the cylinder is formed.

The paper gives information on the compressive stress-strain characteristics of thin sections of cold-rolled stainless steels. The 17 per cent chromium, 7 per cent nickel steels exhibit better compressive properties longitudinal to the direction of rolling, than do the 18 per cent chromium, 8 per cent nickel steels, particularly when the steels are cold rolled to a tensile strength in excess of 150,000 psi. Both steels have better compressive properties transverse to the direction of rolling than longitudinal to the direction of rolling but the difference in the 17 per cent chromium, 7 per cent nickel steels is not so marked as in the 18 per cent chromium, 8 per cent nickel steels. The low-temperature stress-relieving heat treatment (200 to 300 C.) improves the compressive properties of the steels in both directions.

In a recent publication (**1**)[2] data were given on the stress-strain characteristics of cold-rolled steels of the 18 per cent chromium, 8 per cent nickel type in tension as influenced by application of a low-temperature stress-relieving treatment at 200 to 300 C. for periods between 8 and 72 hr. The steels given this heat treatment exhibited a high proportional limit, a high yield strength (0.2 per cent offset), a modulus of elasticity of approximately 28,000,000 psi., increased fatigue resistance, and improved toughness without becoming subject to intergranular corrosion or losing resistance to general corrosion. Also, in confirmation of the work of other investigators, for example, Krivobok *et al.* (2) and Lincoln (3), it was stated that the steels containing 17 to 18 per cent chromium and about 7 per cent nickel with 0.12 per cent carbon could be cold rolled to give high elastic properties in tension without sacrificing ductility, but no information was given on the compressive properties. It is the purpose of the present paper to report on these properties.

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[1] Union Carbide and Carbon Research Laboratories Inc., Niagara Falls, N. Y.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 645.

The use of thin sections of the cold-rolled 18-8 type of steel in lightweight high-strength construction has led to the search for compressive stress-strain data on such sections, but the problem confronted in obtaining these data represents a difficult one. As stated by Aitchison and Tuckerman (4, 5) the strength of structures of thin sections such as in airplane wings and airplane fuselages is generally limited by the strength of certain members carrying compressive loads, and unless such members are designed on the basis of compression it may lead to an unsafe structure or to an uneconomical one. In an effort to make available such information, they developed at the National Bureau of Standards the "pack test" in which an odd number of thin specimens are assembled in a pack and laterally supported with pins while the pack is being tested in compression. Barlow, Stillwell and Ho-Shen Lu (6) described a procedure designated the "plug" method for obtaining the compressive elastic properties of thin-walled sections. This method consists of supporting the section with a cement plug, which restrains the flat sides of the sample and prevents early secondary failure. Recently R. L. Templin of the Aluminum Company of America informed us of a method developed by W. P. Montgomery of the Engineering Dept. of the Vought-Sikorsky Division of United Aircraft Corp. which consists of testing in compression single strip samples that are supported laterally by means of a series of small rolls.

TEST METHOD AND DISCUSSION OF DATA

An investigation was made to find out whether the compressive properties of thin sections could be determined for cold-rolled steels of the 18-8 type by use of cylindrical-shaped samples. Seamless tubes 1 in. in diameter with an 0.035-in. thick wall were obtained and tested. The tubes had been cold drawn to a tensile strength of 131,000 psi. Samples 3.5 in. long having a slenderness ratio of about 10 were prepared from the tubes, after which the ends were machined and ground parallel to within an accuracy of ±0.0002 in. The ends of the cylinders were embedded in Wood's metal on both the inside and outside. On the outside, oversized brass rings were employed to hold the Wood's metal in place. The brass rings reduced the effective column length to approximately 3 in. and permitted the cylinders to be held on a surface grinder while the ends were being ground to the finished dimension. The samples were subjected to compressive stresses using a 60,000-lb. Baldwin-Southwark hydraulic testing machine, and two Huggenberger gages of the metric type were employed to measure the strain. The gage length used was 0.787 in. (2 cm.).

The type of failure obtained in the test is illustrated in Fig. 1 and the stress-strain data are shown in Fig. 2. The compression tests were made on annealed and cold-drawn cylinder samples, which buckled symmetrically to the axis during failure. Stress-strain data obtained on cylinders in tension are included for comparison. Two Huggenberger gages were also used in the tension tests. The data obtained are illustrative of the difference in the stress-strain properties in tension and compression, longitudinal to the direction of cold drawing. The data showed that the yield strength at 0.2 per cent offset[3] in compression is somewhat lower than in tension, particularly in the cold-drawn condition. Other data are given in Table I.

Tests were next made on an 0.035-in. thick 18-8 steel strip that had been cold-rolled to approximately the same tensile strength as the 1-in. outside diameter seamless tubes. Rectangular-shaped sections were cut from the strip and machined to a length of 4.60 in. and a width of about 2.25 in. so that a cylinder could be made. Certain of the sections were annealed and pickled prior to

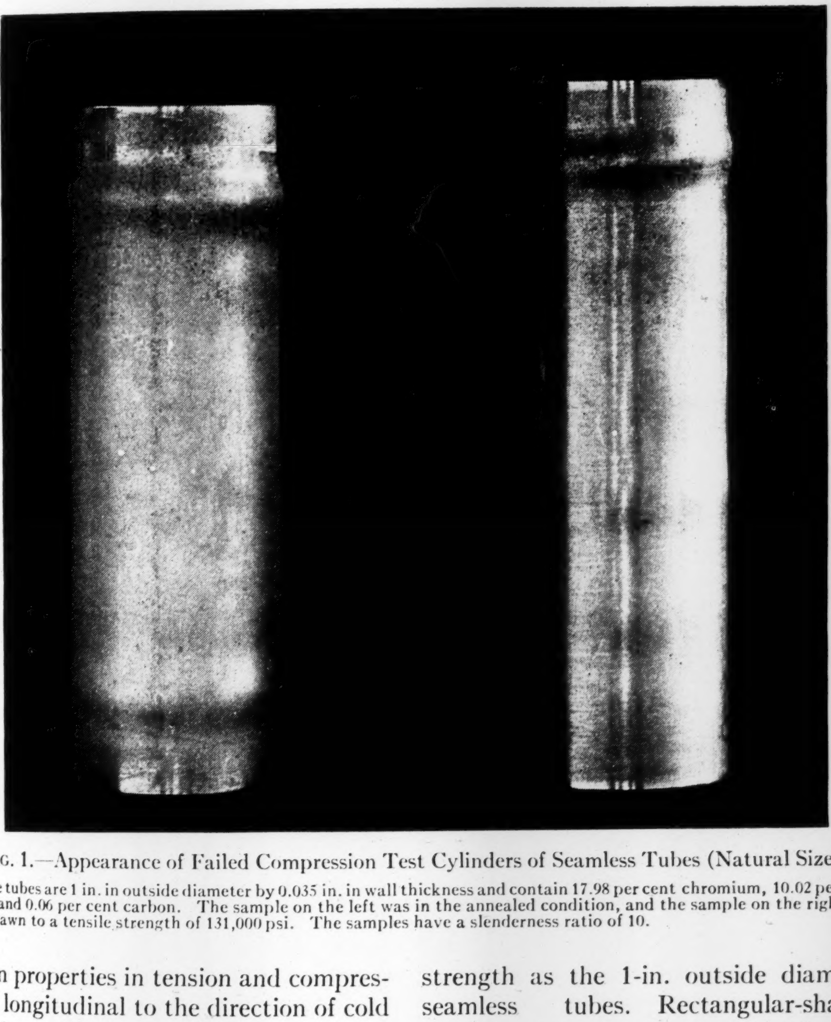

FIG. 1.—Appearance of Failed Compression Test Cylinders of Seamless Tubes (Natural Size).

The tubes are 1 in. in outside diameter by 0.035 in. in wall thickness and contain 17.98 per cent chromium, 10.02 per cent nickel and 0.06 per cent carbon. The sample on the left was in the annealed condition, and the sample on the right was cold drawn to a tensile strength of 131,000 psi. The samples have a slenderness ratio of 10.

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[3] U. S. Navy Department Specification No. 47S21a for Steel, Corrosion-Resisting (18-per cent Chromium and 8-per cent Nickel type), Sheet and Strip, Cold-Rolled, November 1, 1939.

machining, and others were left in the as-cold-rolled condition. Also, certain of the samples were cut longitudinal to the direction of rolling and others transverse to the direction of rolling. The sections were cold formed into cylinders approximately 1.50 in. in diameter by 2.25 in. long, using specially designed bending rolls to obtain a circular cylinder. This diameter of cylinder was selected to facilitate forming.

TABLE I.—DATA OBTAINED IN THE TENSION AND COMPRESSION TESTS OF SEAMLESS TUBES (SEE FIGS. 1 AND 2).

COMPOSITION

Chromium, per cent	17.98
Nickel, per cent	10.02
Manganese, per cent	0.29
Silicon, per cent	0.39
Carbon, per cent	0.06

	Condition of Metal	
	Air Cooled from 1100 C.	As-Cold-Drawn
TENSION		
Proportional limit, psi.	16 800	32 800
Yield strength, psi. (0.2% offset)	38 000	103 000
Tensile strength, psi.	98 000	131 000
Elongation in 6 in., per cent	54	24
Initial tangent *E*, psi.	28 300 000	25 300 000
COMPRESSION		
Proportional limit, psi.	16 800	24 400
Yield strength, psi. (0.2% offset)	31 000	85 000
Buckling stress, psi.	51 000	127 400
Initial tangent *E*, psi.	27 500 000	26 500 000

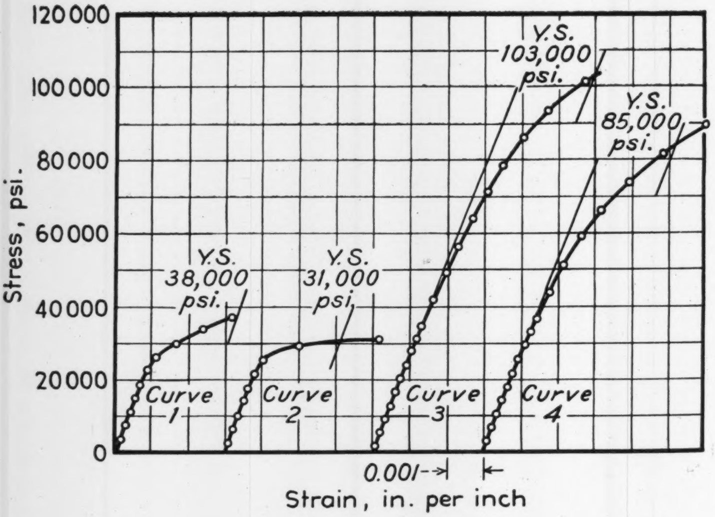

FIG. 2.—Stress-Strain Curves for Seamless Steel Tubes.

The tubes are 1 in. in outside diameter by 0.035 in. in wall thickness and contain 17.98 per cent chromium, 10.02 per cent nickel, and 0.06 per cent carbon. Curves 1 and 3 show tensile properties in the annealed and cold-drawn conditions. Curves 2 and 4 show compression properties (samples in Fig. 1) in the annealed and cold-drawn conditions. These properties were of necessity determined longitudinal to the direction of cold drawing.

The cylinders were placed on a close-fitting soft steel arbor, and clamped lightly, after which the longitudinal seam was sealed using soft solder to form a continuous cylinder. The arbor containing the soldered cylinder was set up between centers on a lathe, and the cylinder machined to exactly 2 in. in length, allowing the tool bit to cut into the steel arbor to remove the burr that formed. As in the case of the seamless tube samples, the ends of the soldered cylinder were embedded in Wood's metal to a depth of about $\frac{1}{4}$ in. using an oversize brass ring on the outside to retain the low-melting alloy. The ends of the cylinder were made to clear the

Wood's metal so as not to interfere with testing. After grinding, the uniformity in length of the cylinder was checked with a surface gage, and if a total difference of more than 0.0004 in. was found, the ends were reground. Four checks on the length of the cylinder were made, about 90 deg. apart on the circumference of the ends. The finished cylinder, which was now ready for the compression test, had a slenderness ratio of about 5, a diameter to thickness ratio of about 40 and an effective column length of about 1.5 in. These values were adhered to in designing the sample in order to obtain a high degree of stability while it was being stressed in the plastic range.

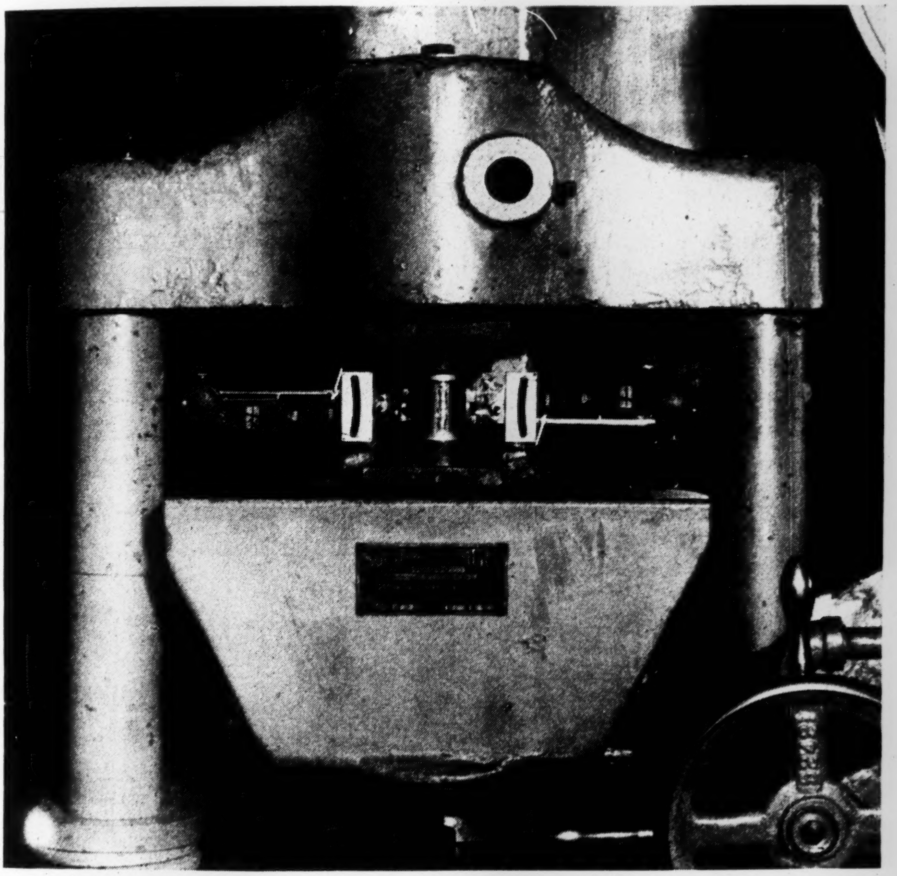

FIG. 3.—Showing the Assembly Used in Making the Compression Test on the Cold-Formed and Soldered Cylinder.

In making the compression test, two Huggenberger extensometers of the metric type having a multiplication ratio of 1175 were used to measure the strain. The knife edges of the gages were placed opposite each other and at about 90 deg. from the soldered seam. The cylinder with the attached Huggenberger gages was placed in the testing machine, as shown in Fig. 3, between two hardened steel blocks ground flat. A small piece of blotting paper was inserted between the bottom plate and the testing machine to prevent slippage. To offset trouble due to the heads of the machine not being parallel, a plaster of Paris shim was placed between the upper block and the head of the machine. The shim, the mixture which is approximately 50 g. of plaster of Paris and 20 ml. of water, was spread uniformly between two pieces of tracing paper before being placed between the steel block and the head of the machine. A small load of 100 lb. was

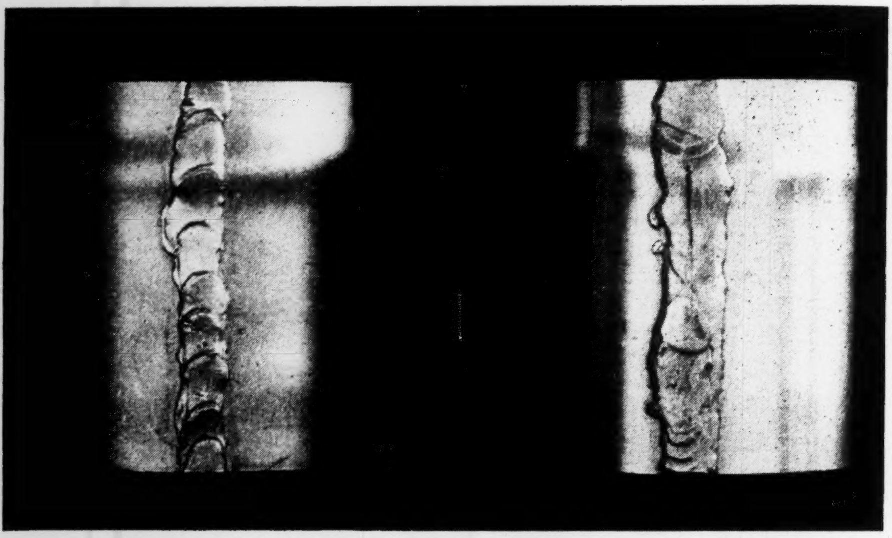

FIG. 4.—Cold-Formed Cylinders After Failure in the Compression Test (Natural Size).

The cylinders were prepared longitudinal to the direction of rolling from 0.035 in. thick strip of a steel containing 18.45 per cent chromium, 8.79 per cent nickel, and 0.10 per cent carbon. The cylinder on the left was formed from an annealed sample and the one on the right from an as-cold-rolled sample. Cylinders of the same steel prepared to test the properties transverse to the direction of rolling also failed symmetrical to the axis.

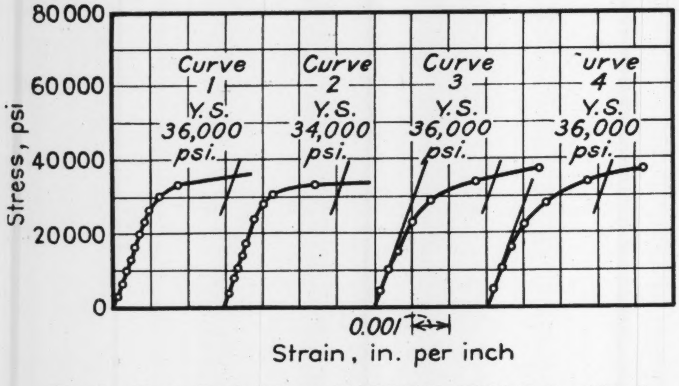

FIG. 5.—Stress-Strain Curves for 0.035 in. Thick Strip of Steel.

The steel contains 18.45 per cent chromium, 8.79 per cent nickel, and 0.10 per cent carbon in the annealed condition. Curves 1 and 2 are for tension properties in the longitudinal and transverse directions to rolling, respectively. Curves 3 and 4 are for compression properties in the longitudinal and transverse directions to rolling, respectively.

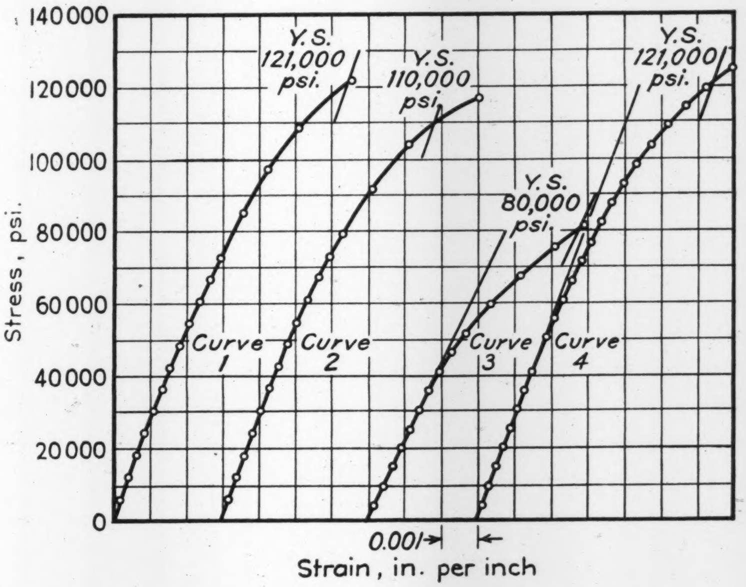

FIG. 6.—Stress-Strain Curves for 0.035 in. Thick Strip of Steel.

The steel contains 18.45 per cent chromium, 8.79 per cent nickel and 0.10 per cent carbon and is cold rolled to a tensile strength of 139,300 psi. Curves 1 and 2 are for tensile properties in the longitudinal and transverse directions to rolling, respectively. Curves 3 and 4 are for compressive properties in the longitudinal and transverse directions to rolling, respectively.

TABLE II.—DATA OBTAINED IN THE TENSION AND COMPRESSION TESTS (SEE FIGS. 4, 5, AND 6).

COMPOSITION

Chromium, per cent	18.45
Nickel, per cent	8.79
Manganese, per cent	0.50
Silicon, per cent	0.45
Carbon, per cent	0.10

	Condition of Metal			
	Air Cooled from 1100 C.		As-Cold-Rolled	
	Longitudinal[a]	Transverse[a]	Longitudinal[a]	Transverse[a]
TENSION				
Proportional limit, psi.	13 300	16 600	21 000	40 000
Yield strength, psi. (0.20% offset)	36 000	34 000	121 000	110 000
Tensile strength, psi.	94 500	93 700	139 300	138 500
Elongation in 2 in., per cent	61	61	22	17
Initial tangent E, psi.	29 000 000	29 000 000	27 000 000	29 000 000
COMPRESSION				
Proportional limit, psi.	11 000	11 000	23 700	36 900
Yield strength, psi. (0.20% offset)	36 000	36 000	80 000	121 000
Buckling stress, psi.	50 250	50 000	120 400	145 000
Initial tangent E, psi.	28 000 000	28 000 000	22 000 000	27 000 000

[a] To direction of rolling.

applied to the assembly for about 20 min. to allow the plaster of Paris to set before the test was started. The magnitude of this load depends on the area of the sample.

In testing the 0.035-in. thick wall

cylinders, the initial strain reading was recorded at a load of 100 lb. Load increments of 100 to 200 lb. were then applied to a stress slightly beyond the proportional limit, after which 800 and 1600-lb. load increments were used. At a point somewhat below the yield strength at 0.2 per cent offset, the increments of load were again reduced to about 200 lb. At this point the rate of loading was relatively slow, and sufficient time was allowed after each load to be sure that the correct strain value was obtained. Loading was continued until the yield strength had been slightly exceeded. The Huggenberger extensometers were then removed and the loading was increased further until failure occurred by buckling.

This procedure was used in making tests on the previously discussed 18-8 steel strip and the results are described in Figs. 4, 5, and 6, and Table II.

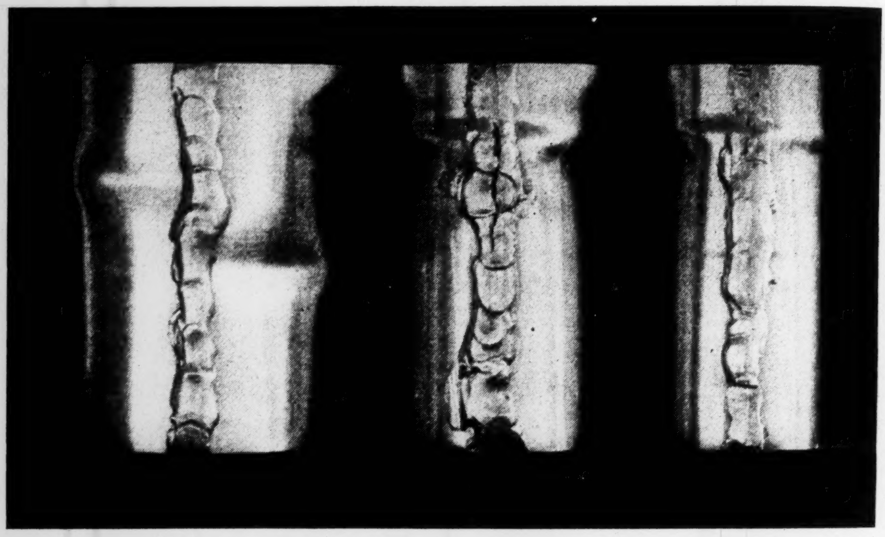

FIG. 7.—Showing the Failed Compression Test Cylinders of 0.02 in. Thick Strip Longitudinal to the Direction of Rolling(Natural Size).

The steel contains 17.90 per cent chromium, 6.72 nickel, and 0.11 per cent carbon cold rolled to a tensile strength of about 220,000 psi. The cylinder at the extreme left had a 1.5-in. diameter and exhibited nonuniform local failure as did the 1-in. diameter cylinder represented by the middle photograph. However, the 0.8-in. diameter cylinder at the extreme right failed uniformly and is considered satisfactory.

Figure 4 shows that the cold-formed cylinders with soldered joint failed symmetrically to the axis in a manner similar to that of the cold-drawn seamless tubes shown in Fig. 1. The stress-strain data in Figs. 5 and 6 and Table II of tests made longitudinal to the direction of rolling confirmed those obtained on the seamless tubes in the annealed and cold-drawn conditions, indicating that the cylinder cold formed from the strip was properly designed with respect to the slenderness and diameter-to-thickness ratios. It was of further interest to note that the soldered joint in the cylinder functioned satisfactorily.

As before, tension data are given in Figs. 5 and 6 and Table II for the sake of comparison. In these tests the slope of the modulus line was determined using two Huggenberger gages, but the yield strength at 0.2 per cent offset was determined using the Berry gage. It was again observed that in the annealed condition tension and compressive properties were similar, but after cold rolling the compressive properties longitudinal to the direction of rolling were inferior to the tensile properties in the same direction. On the other hand, the tensile and compressive properties transverse to the direction of rolling were quite similar, the compressive properties transverse to the direction of rolling being decidedly superior to those longitudinal.

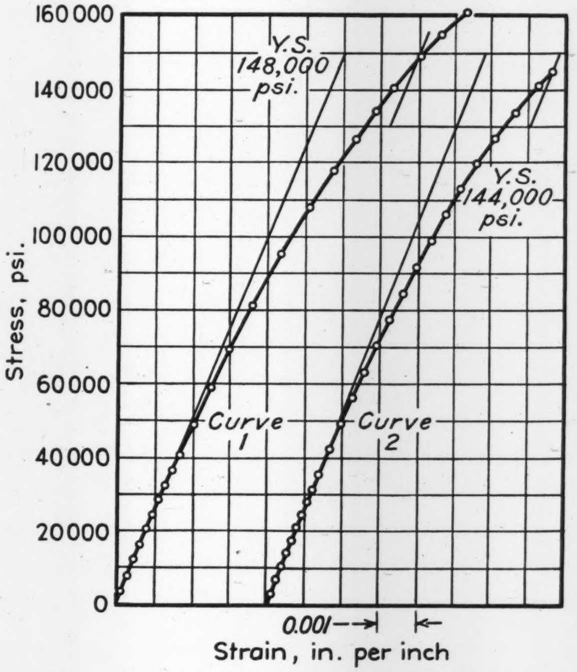

FIG. 8.—Compression Stress-Strain Curves for 0.02 and 0.01 in. Thick Strip of Steel.

The steel contains 17.90 per cent chromium, 6.72 per cent nickel, and 0.11 per cent carbon cold-rolled to a tensile strength of 220,000 psi. Curve 1 gives the data obtained on the 0.8-in. diameter cylinder of 0.02-in. thick strip, and curve 2 gives the data for the 0.4-in. diameter cylinder from the 0.01-in. thick strip. The samples were tested in the as-cold-rolled condition, longitudinal to the direction of rolling.

The investigation was continued using 0.02 and 0.01-in. thick strip of a steel containing 17.90 per cent chromium, 6.72 per cent nickel, and 0.11 per cent carbon cold rolled to a tensile strength of about 220,000 psi. It was necessary to construct two additional sets of bending rolls to form cylinders from these thinner strips. Cylinders having diameters of about 1.50, 1, and 0.80 in. were made from the 0.02-in. thick strip and tested to determine the compressive properties in both directions to rolling. Tension tests were made on the strip, but for the sake of brevity only the data secured in the longitudinal direction in the compression tests will be discussed.

As shown in Fig. 7, the 1.5-in. diameter cylinder failed locally near the center of the cylinder. Also the failure was not symmetrical to the axis of the cylinder. A similar result was obtained with the 1-in. diameter cylinder. Neither of these failures resembled those obtained with the seamless tubes or the cold-formed 1.5-in. diameter cylinder of the 0.035-in. thick strip, and the tests were considered unsatisfactory. The failure occurring with the 0.8-in. diameter cylinder was quite different. This cylinder failed symmetrically and in a manner similar to that of the seamless tube. This test was considered satisfactory, and showed that with a slenderness ratio of about 7, the cylinder should have a maximum diameter-to-thickness ratio of about 40 to avoid local unsymmetrical failure. This was subsequently proven in the tests on the 0.01-in. thick strip of the same steel. It was necessary to reduce the diameter of the cylinder from the 0.01-in. thick strip to approximately 0.40 in. to avoid the same difficulty. The over-all length of the cylinder was reduced to 1.5 in., and with the brass ring attached the effective column length was 1.25 in. As before, the gage length was 0.787 in. (2 cm.).

TABLE III.—DATA OBTAINED IN THE COMPRESSION TESTS OF FIG. 4.

COMPOSITION OF STRIP

Chromium, per cent	17.90
Nickel, per cent	6.72
Manganese, per cent	0.56
Silicon, per cent	0.27
Carbon, per cent	0.11

	0.02 in. Thick Strip. As Cold Rolled. Longitudinal to the Direction of Rolling	0.01 in. Thick Strip. As Cold Rolled. Longitudinal to the Direction of Rolling
Proportional limit, psi.	32 000	30 000
Yield strength, psi. (0.20% offset)	148 000	144 000
Buckling stress, psi.	209 000	213 000
Initial tangent *E*, psi.	25 000 000	26 700 000

The data from the satisfactory compression tests on the 0.8-in. diameter cylinder of 0.02-in. thick strip, and the 0.4-in. diameter cylinder of the 0.01-in. thick strip are given in Fig. 8 and Table III. These are of interest because they show that if the steel is cold rolled to the same tensile strength, the compressive properties longitudinal to the direction of rolling are similar in strips of different thicknesses.

The dimensions of the cylinders required to test strips of different thicknesses satisfactorily is of interest. The cylinders from the 0.035-in. thick strip should have a diameter of about 1.5 in. and an over-all length of about 2 in.; the cylinders from the 0.02-in. strip should have a diameter of 0.80 in. and a similar length; the cylinders from the 0.01-in. thick strip should have a diameter of about 0.4 in. and an over-all length of about 1.5 in. Cylinders of these dimensions with the brass rings attached have a slenderness ratio between 5 and 10 and a maximum diameter to thickness ratio of about 40. It is probable that the over-all length of the cylinders can be increased somewhat, but it is doubtful whether the diameter-to-thickness ratio can be changed materially without encountering trouble.

As previously stated, the elastic properties of the cold-rolled stainless steels are improved by the low-temperature heat treatment (200 to 300 C.). So, chromium-nickel steels of the 18-8 type containing different percentages of chromium, nickel, and carbon were made and tested in compression to determine the influence of this treatment as well as of the composition. All the data obtained cannot be reported but enough of them will be shown with corresponding tension data to illustrate the important differences. The compression tests were conducted on cylinders of cold-rolled strip approximately 0.035 in. thick. The respective cylinders, which had a diameter of 1.5 in. and were 2 in. long with an effective column length of 1.5 in., were prepared as previously described, with one exception. In the preparation of the low-temperature stress-relieved samples, the cylinders were cold formed as usual, stress-relieved, and then soldered followed by machining and grinding to the finished dimensions. The samples were tested

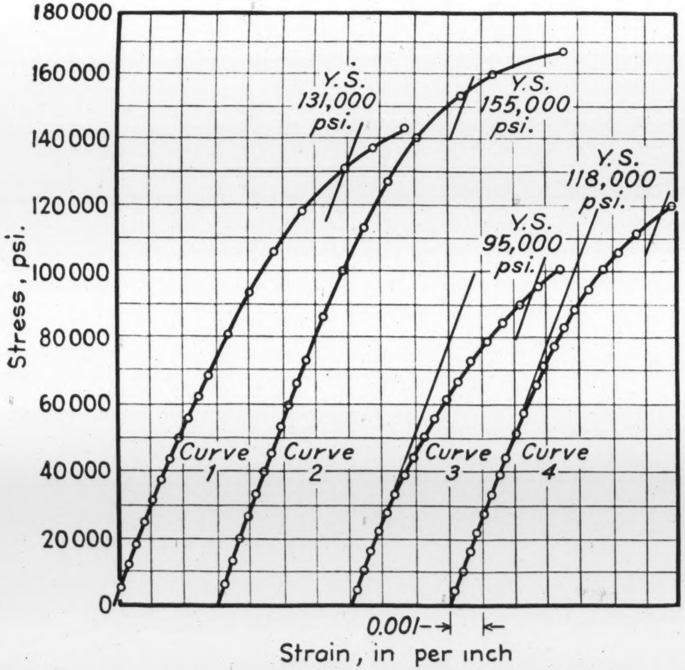

FIG. 9.—Stress-Strain Curves for 0.035 in. Thick Strip of Steel No. 13.

Reduced 35 per cent by cold rolling. Tested longitudinal to the direction of rolling.

Tension

Curve 1	As-cold-rolled
Curve 2	Heat-treated at 200 C.

Compression

Curve 3	As-cold-rolled
Curve 4	Heat-treated at 200 C.

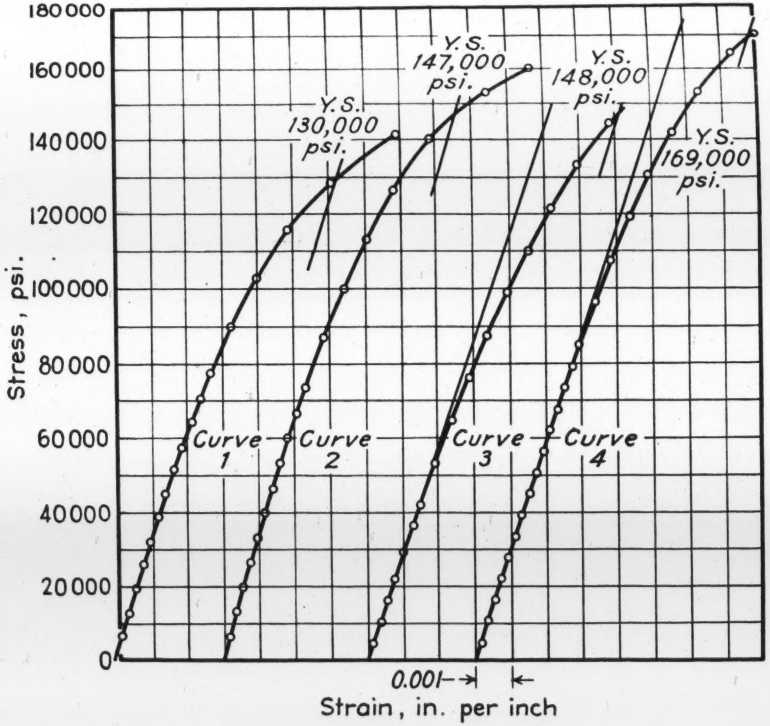

FIG. 10.—Stress-Strain Curves for 0.035 in. Thick Strip of Steel No. 13.

Reduced 35 per cent by cold rolling. Tested transverse to the direction of rolling.

Tension

Curve 1	As-cold-rolled
Curve 2	Heat-treated at 200 C.

Compression

Curve 3	As-cold-rolled
Curve 4	Heat-treated at 200 C.

using an 0.787-in. (2-cm.) gage length, in the annealed condition, in the as-cold-rolled condition, and after cold rolling and stress-relieving at 200 to 300 C. They were tested both longitudinal and transverse to the direction of rolling. The data given in the accompanying figures and tables show the relationship between the tensile and compressive properties of the different steels. The tension data were obtained using a standard A.S.T.M. ½-in. flat tension specimen with a 2-in. gage length. As previously stated, the yield strength at 0.2 per cent offset in tension was determined using the Berry gage, but as shown in the tables, the initial tangent modulus in tension was determined with two Huggenberger gages the same as those employed in the compression test using an 0.787-in. (2-cm.) gage length.

TABLE IV.—DATA OBTAINED IN TENSION AND COMPRESSION TESTS ON 0.035-IN. THICK STRIP SHOWN IN FIGS. 9 AND 10.

Heat	Composition, per cent					Condition of Metal[a]	Cold Reduction, per cent	Direction to Rolling[b]	Tension[c]					Compression[d]			
	Chromium	Nickel	Manganese	Silicon	Carbon				Initial Tangent E, in million psi.	Proportional Limit, psi.	Yield Strength, 0.2% offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Initial Tangent E, in million psi.	Proportional Limit, psi.	Yield Strength, 0.2% offset, psi.	Buckling Stress, psi.
No. 13	18.45	8.79	0.50	0.55	0.10	A	None	L	29	13 300	36 000	94 500	61	28	11 000	36 000	50 250
						A	None	T	29	16 600	34 000	93 700	61	28	11 000	36 000	50 000
	18.45	8.79	0.50	0.55	0.10	1	35	L	26	46 600	131 000	155 300	15	26	17 100	95 000	151 800
						2	35	L	27	56 600	155 000	173 400	11	27	47 600	118 000	158 000
						1	35	T	30	53 000	130 000	166 200	11	27	42 000	148 000	183 000
						2	35	T	31	53 000	147 000	178 700	10	29	72 000	169 000	193 000
	18.45	8.79	0.50	0.55	0.10	1	50	L	26	37 000	155 000	177 400	6	21	41 000	112 000	155 200
						2	50	L	27	54 000	170 000	187 000	5	26	43 000	126 000	168 200
						1	50	T	30	41 500	147 000	189 200	8	28	51 000	166 000	166 700
						2	50	T	31	53 000	164 000	198 300	6	29	65 000	183 000	211 000

[a] 1 = as cold rolled, 2 = heated 72 hr. at 200 C. and air cooled.

[b] L = longitudinal to direction of rolling, T = transverse to direction of rolling.

A = annealed.

[c] Berry gage tests. Value for *E* based on Huggenberger gage data.

[d] The 1.5-in. diameter cylinder tested had a slenderness ratio of about 5 and a diameter-thickness ratio of about 40

The tension and compression data on the 18-8 steels are shown in Figs. 9 and 10 and in Table IV. These showed that although the annealed longitudinal and transverse properties were quite similar, a marked difference existed after cold rolling. In tension the longitudinal properties were slightly superior to the transverse properties, but in compression the transverse properties were superior to the longitudinal properties. Increasing the degree of cold rolling from 35 to 50 per cent did not greatly improve the compressive properties longitudinal to the direction of rolling even though the strength in tension was materially enhanced. In every instance the stress-relieving heat treatment at about 200 C. markedly improved the elastic properties of the material in both directions in both tension and compression. Thus, these tests showed the importance of the conclusion of Aitchison and Tuckerman (4) that it was unwise to design compression members on the basis of tensile stress-strain curves.

The data for the 17 per cent chromium, 7 per cent nickel steel and 17 per cent chromium, 6 per cent nickel steel are given in Figs. 11, 12, 13, and 14 and Table V, and are quite different from those of the cold-rolled 18-8 steel. When cold reduced to the same extent

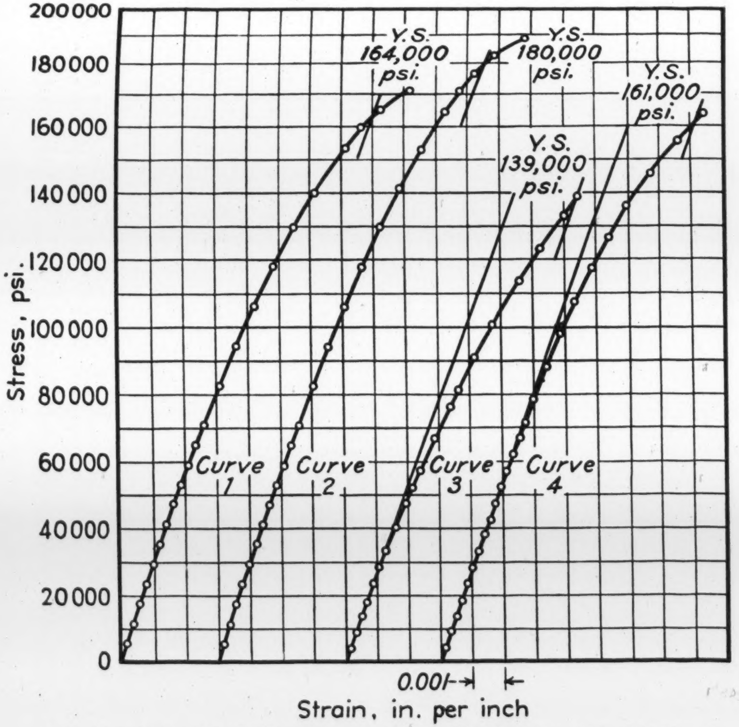

FIG. 11.—Stress-Strain Curves for 0.035 in. Thick Strip of Steel E717.

Reduced 35 per cent by cold rolling. Tested longitudinal to the direction of rolling

Tension	
Curve 1	As-cold-rolled
Curve 2	Heat-treated at 300 C.
Compression	
Curve 3	As-cold-rolled
Curve 4	Heat-treated at 300 C.

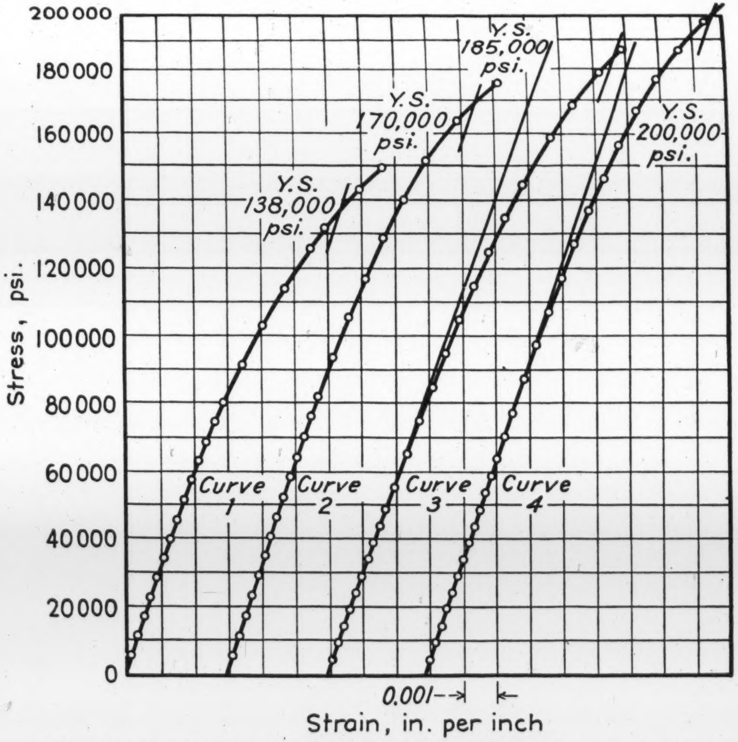

FIG. 12.—Stress-Strain Curves for 0.035 in. Thick Strip of Steel E717.

Reduced 35 per cent by cold rolling. Tested transverse to the direction of rolling.

Tension	
Curve 1	As-cold-rolled
Curve 2	Heat-treated at 200 C.
Compression	
Curve 3	As-cold-rolled
Curve 4	Heat-treated at 200 C.

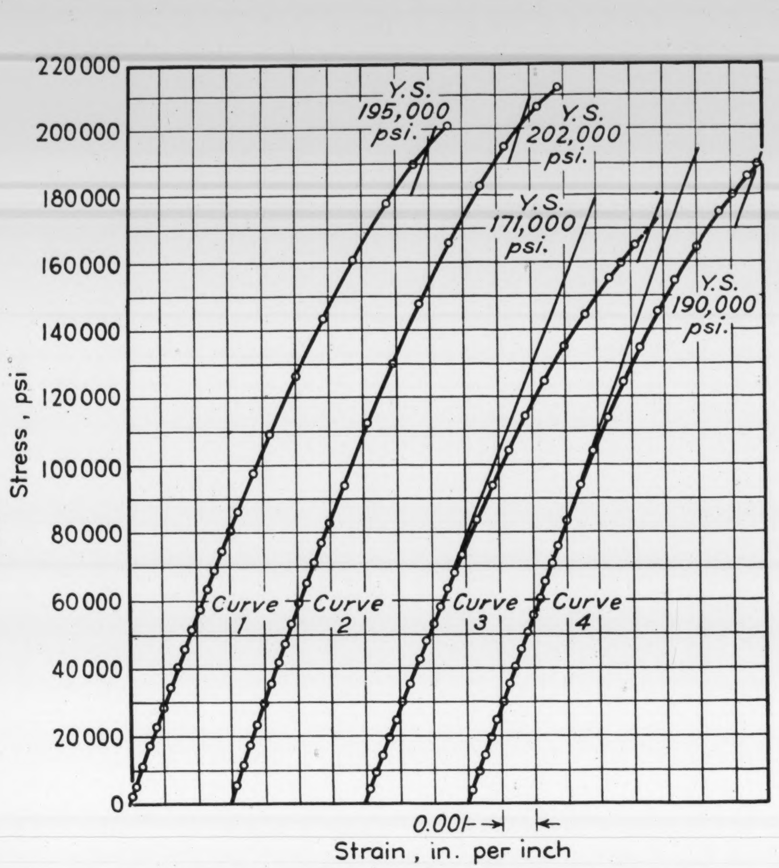

FIG. 13.—Stress-Strain Curve for 0.035 in. Thick Strip of Steel E850.

Reduced 35 per cent by cold rolling. Tested longitudinal to the direction of rolling.

Tension

Curve 1 .... As-cold-rolled
Curve 2 .... Heat-treated at 200 C.

Compression

Curve 3 .... As-cold-rolled

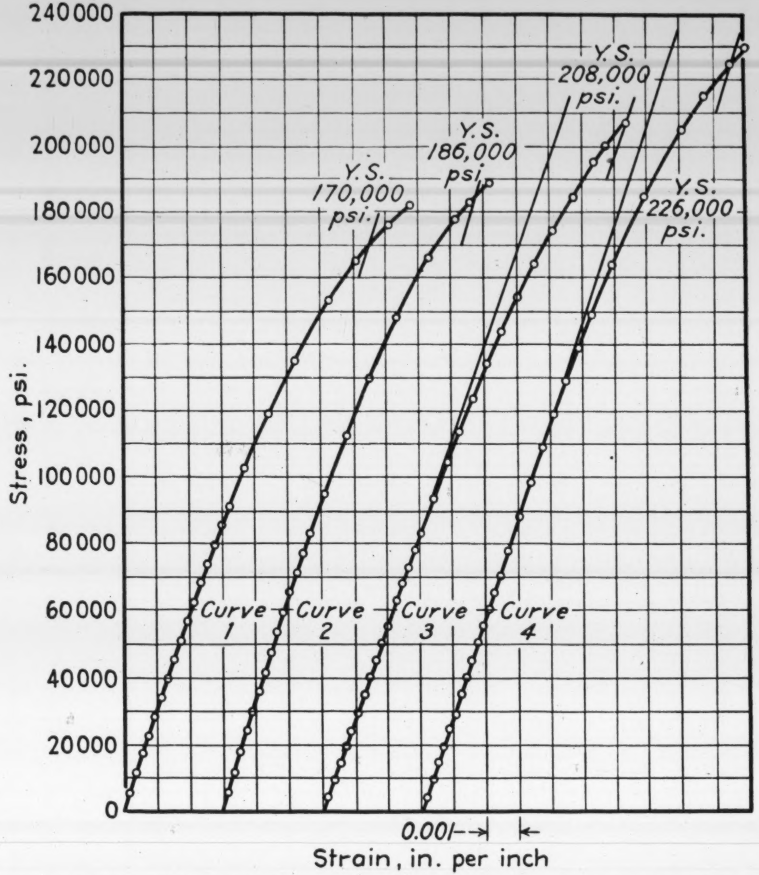

FIG. 14.—Stress-Strain Curves for 0.035 in. Thick Strip of Steel E850.

Reduced 35 per cent by cold rolling. Tested transverse to the direction of rolling.

Tension

Curve 1 .... As-cold-rolled
Curve 2 .... Heat-treated at 200 C.

Compression

Curve 3 .... As-cold-rolled

(35 per cent), these two steels exhibited better stress-strain characteristics in tension and compression than did the 18-8 steel. Even though the 17-7 and 17-6 steels had better elastic properties in the transverse direction to rolling in compression than in the longitudinal direction, it should be noted that the two steels had good properties in both directions to rolling. Moreover, the low-temperature stress-relieving heat treatment at 200 to 300 C. was very effective in improving the stress-strain characteristics of the two steels. This is amply illustrated by the fact that longitudinal to the direction of rolling, the 17-7 steel had a yield strength (0.2 per cent offset) in compression after stress-relieving, of 161,000 psi., and the 17-6 steel a yield strength of 190,000 psi.

Additional evidence revealing the improvement obtained by application of the low-temperature heat treatment is shown by Fig. 15, which gives the tangent modulus curves before and after the treatment in tension and compression both transverse and longitudinal to the direction of rolling. These curves were prepared from the data on the 17-7 steel (E717) using the stress-strain values between the observed proportional limit and the yield strength at 0.2 per cent offset. The curves are more or less self-explanatory, but it should be mentioned to illustrate the stiffening effect produced by the heat treatment that for certain stresses there is a difference in the modulus of elasticity of the as-cold-rolled material and the stress-relieved material of between 3,000,000 and 5,000,000 psi. This should leave little doubt of the advisability for stress-relieving the as-cold-rolled metal in order to obtain the most rigid lightweight high-strength structures.

TABLE V.—DATA OBTAINED IN TENSION AND COMPRESSION TESTS ON 0.035 IN. THICK STRIP SHOWN IN FIGS. 11, 12, 13, AND 14.

Heat	Composition, per cent					Condition of Metal[a]	Cold Reduction, per cent	Direction to Rolling[b]	Tension[c]					Compression[d]			
	Chromium	Nickel	Manganese	Silicon	Carbon				Initial Tangent E, in million psi.	Proportional Limit, psi.	Yield Strength, 0.2% offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Initial Tangent E, in million psi.	Proportional Limit, psi.	Yield Strength, 0.2% offset, psi.	Buckling Stress, psi.
No. E572	17.20	7.23	1.28	0.43	0.11	A	None	L	29	14 000	33 000	117 800	68	28	12 000	40 000	57 800
						A	None	T	29	15 700	33 000	113 500	62	29	12 000	44 000	57 400
No. 16A	17.16	7.40	0.58	0.30	0.12	1	25	L	..	....	121 000	177 900	30	27	16 000	98 000	136 000
						2	25	L	28	45 500	133 000	172 900	26	27	36 000	116 000	159 000
						1	25	T	..	....	103 000	175 100	27	28	27 000	137 000	168 000
						2	25	T	29	47 000	127 000	171 000	25	29	55 000	143 000	171 000
No. E717	17.15	7.17	1.32	0.34	0.11	1	35	L	26	44 000	164 000	196 000	15	26	29 000	139 000	208 500
						3	35	L	27	53 000	180 000	198 000	14	27	62 000	161 000	210 300
						1	35	T	30	46 400	138 000	201 000	10	27	47 000	185 000	214 300
						2	35	T	30	61 000	170 000	202 000	10	29	78 000	200 000	218 500
No. E850	17.45	6.20	1.31	0.34	0.13	1	35	L	28	46 000	195 000	229 500	14	26	41 000	171 000	226 300
						2	35	L	29	58 000	202 000	225 500	12	28	74 000	190 000	231 500
						1	35	T	30	50 000	170 000	224 500	10	28	69 000	208 000	239 500
						2	35	T	31	60 000	186 000	226 800	9	28	119 000	226 000	250 500

[a] 1 = as cold rolled, 2 = heated 72 hr. at 200 C. and air cooled, 3 = heated 24 hr. at 300 C. and air cooled.
[b] L = longitudinal to direction of rolling, T = transverse to direction of rolling.
A = annealed.
[c] Berry gage tests. Value for E based on Huggenberger gage data.
[d] The 1.5-in. diameter cylinder tested had a slenderness ratio of about 5 and a diameter-to-thickness ratio of 40.

SUMMARY

This paper describes a method for determination of the stress-strain characteristics of thin sections of cold-rolled stainless steels in compression. The method is applicable to other materials and is referred to as the *"Cylinder Method"* because of the type of specimen employed. With this method the yield strength at 0.2 per cent offset is readily determined for sections of cold-rolled stainless steels as thin as 0.01 in. and as thick as 0.06 in., provided the slenderness ratio is under 15 and the diameter-to-thickness ratio does not greatly exceed 40. The data obtained on the cold-formed and soldered cylinder are comparable to those secured on seamless cylinders. In addition the cylinder method permits the transverse stress-strain characteristics to be determined.

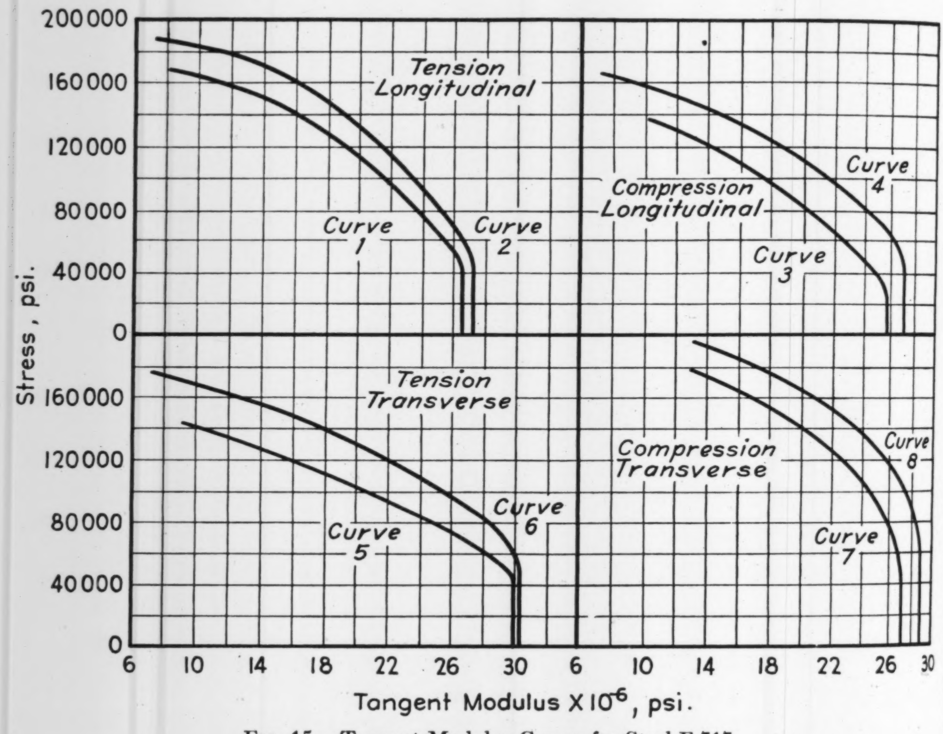

FIG. 15.—Tangent Modulus Curves for Steel E 717.

Reduced 35 per cent by cold rolling to a tensile strength of 196,000 psi.

Curve 1	As-cold-rolled
Curve 2	Heat-treated at 300 C.
Curve 3	As-cold-rolled
Curve 4	Heat-treated at 300 C.
Curve 5	As-cold-rolled
Curve 6	Heat-treated at 200 C.
Curve 7	As-cold-rolled
Curve 8	Heat-treated at 200 C.

The actual data for the cold-rolled chromium-nickel steels show that longitudinal to the direction of rolling the tensile stress-strain characteristics are superior to those in compression. Transverse to the direction of rolling the compressive properties are superior to the tensile properties, and as would be inferred, the transverse compression properties are superior to the longitudinal compression properties.

The best combination of properties is obtained in steels containing 17 to 18 per cent chromium and about 7 per cent nickel, especially when the steels are cold-rolled to high strength. These steels have high ductility and excellent tension and compressive stress-strain characteristics both longitudinal and transverse to the direction of rolling. The stress-strain characteristics of the steels are markedly improved by the low-temperature heat treatment at 200 to 300 C. for 8 to 72 hr.

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ADDITION OF BISMUTH FOR PRODUCING FREE-MACHINING STAINLESS STEELS

By H. Pray,[1] R. S. Peoples,[2] and F. W. Fink[2]

Synopsis

The addition of small amounts of bismuth (0.1 to 0.5 per cent) to the corrosion-resistant stainless alloys results in a remarkable and useful increase in their machinability with no detriment to, and in some cases, an improvement in their corrosion resistance. The paper describes laboratory data relative to the effect of bismuth on the machinability, corrosion resistance to a variety of media and conditions, physical properties at ordinary and high temperature, galling resistance and the hot working properties of several of the more common stainless alloy types. The general metallurgy of the bismuth-containing alloys is discussed. The laboratory data and results are substantiated by plant and service experience.

Machine shops always want better machining properties in the steels they handle, and the shops that have to handle corrosion-resistant steels are no exception.

Recognizing the benefits to be gained through improved machinability, the Alloy Casting Research Institute[3] instructed Battelle Memorial Institute to study the problem with particular relation to castings, making it clear, however, that it was essential that improved machinability would only be acceptable if it were attained without material sacrifice in corrosion resistance or mechanical properties.

The evaluation in the laboratory of machinability has been reputed to be difficult and inconclusive, and this is broadly true when very slight differences in machinability have to be evaluated. It is also true that different alloys fall into somewhat different orders of machinability, depending on the particular type of machining operation used as the criterion. Yet, between two alloys of such really different machinability that any machinist would recognize the difference, laboratory tests can likewise recognize the difference and express it in roughly quantitative values. This was true in the case of lead-bearing free-machining steel, the comparative sawability and drillability tests made in this laboratory by the methods described by Nead, Sims, and Harder,[4] gave data that were fully corroborated by later commercial experience. Hence the laboratory tests for machinability used in the present study were essentially the same sawing and drilling tests described by Nead, Sims and Harder, the reference material of 100 sawability or drillability index being, in each case, an addition-free lot of whatever corrosion resistance casting composition was being studied.

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[4] J. H. Nead, C. E. Sims, and O. E. Harder, "Properties of Some Free-Machining Lead-Bearing Steels," *Metals and Alloys*, Vol. 10, pp. 68–73, 109–114 (1939).

These indices express the ratios of the time necessary to do the same cutting on the reference alloy and the alloy tested. If it takes 200 sec. to make a certain cut or certain drilling in the reference alloy and the alloy under test takes but 150 sec., its index is 133; if it takes 250 sec., the index is 80.

The sawability and drillability times were determined on 25 per cent chromium, 12 per cent nickel, and 19 per cent chromium, 9 per cent nickel castings as reference alloys and on castings of similar compositions to which S, Se, Mo, Cu, P, Ag, Pb, Bi, and a few other elements, alone or in combination were added. Some of these were helpful. Sulfur or selenium, alone or in combination with other elements, of course, improved machinability, but affected corrosion resistance to a degree not permissible under the aims of this investigation. Silver gave erratic results in the laboratory, possibly due to difficulties in incorporating the silver into the melt in uniform suspension. Lead was helpful, but its introduction under the conditions obtaining in foundries entails some difficulties. When bismuth was tried its beneficial effect on machinability was at once apparent. Plant trials showed it to be entirely feasible to introduce bismuth under commercial conditions, so the investigation then settled down to a comprehensive study of the effects of bismuth.

Table I gives machinability data for a variety of compositions and shows that bismuth is effective in increasing machinability of the corrosion-resistant alloys as a class. The laboratory indications were corroborated by the opinion of the Battelle machine shop and likewise by the machine shops of Alloy Casting Institute members handling commercially produced material. The surface finish obtainable with carboloy tools in finish turning at 44, 85, and 118 ft. per min. was undistinguishable from that of the bismuth-free alloys.

MECHANICAL PROPERTIES

Charpy impact tests, standard key-hold notch, gave the data in Table II.

Short-time tension tests at room temperature and at 1800 F. gave the data of Table III.

Room temperature properties are not materially altered by presence of bismuth. Strength at 1800 F. is unaffected, but ductility is decreased.

Hot Working:

Hot working of bismuth-containing 12 per cent chromium; 19 Cr, 9 Ni; and 25 Cr, 12 Ni was attempted. The 12 per cent chromium alloy with 0.10 per cent bismuth forged and rolled well. Both 19 Cr, 9 Ni, and 25 Cr, 12 Ni with about 0.25 per cent bismuth forged satisfactorily at 1800 to 1900 F., but showed a strong tendency to crack at 2150 F. Small rods have been swaged from hot-rolled rounds. Rolling and forging operations have been successfully accomplished in the plants of A.C.R.I. member companies.

Galling:

The galling propensities of these alloys are of some importance in valves, gears, pumps, etc. In the Amsler machine, as it is used for galling and seizure tests, two disks are rotated together, with the periphery of one moving at a different speed from that of the other. This results in a slip, which in the case of the tests to be cited, amounts to 32.5 per cent. After lapping, the specimens were cleaned while in place on the machine, and then tested, using a mixture of

TABLE I.—MACHINABILITY DATA.

All heats water quenched from 2050 F. except those noted.

Heat	Intended Composition, per cent[a]			Actual Bismuth Content, per cent	Drill Index	Saw Index[b]	
	Chromium	Nickel	Other			Compared to 19 Cr, 9 Ni or 25 Cr, 12 Ni	Compared to Materials of Same Analysis Without Bi
CAST ALLOYS (FIRST SERIES)							
No. 5162	18	8	....	....	100	100	..
No. 5163	25	12	....	....	100	100	..
No. 5238	18	8	....	0.12	193	130	..
No. 5239	25	12	....	0.11	132	112	..
No. 5240	25	12	....	0.13	147	133	..
No. 5241	25	12	....	0.25	142	130	..
No. 5242	25	12	....	0.37	265	139	..
CAST ALLOYS WITH VARYING BISMUTH CONTENT (SECOND SERIES)							
No. 5346	19	9	....	0.00	100	100	..
No. 5358	19	9	....	0.04	238	110	..
No. 5350	19	9	....	0.08	183	108	..
No. 5352	19	9	....	0.13	190	121	..
No. 5353	19	9	....	0.14	212	127	..
No. 5391	19	9	....	0.16	188	137	..
No. 5367	19	9	....	0.19	300	141	..
No. 5392	19	9	....	0.19	313	134	..
No. 5396	19	9	....	0.24	204	142	..
No. 5393	19	9	....	0.27	443	159	..
No. 5395	19	9	....	0.32	411	161	..
No. 5347	25	12	....	0.00	100	100	..
No. 5355	25	12	....	0.05	120	103	..
No. 5356	25	12	....	0.09	359	176	..
No. 5357	25	12	....	0.13	461	147	..
No. 5398	25	12	....	0.34	421	210	..
LARGE TEST BLOCK CASTINGS							
No. 5455-1	19	9	....	0.00	100	100	..
No. 5455-2	19	9	....	0.20	173	123	..
No. 5455-3	19	9	....	0.26	206	120	..
CAST ALLOYS WITH OTHER ADDITIONS WITH BISMUTH							
No. 5552	19	9	....	0.00	..	100	100
No. 5552 (As-cast)	19	9	....	0.00	..	100	..
No. 5572 (As-cast)	19	9	....	0.20	..	138	..
No. 5553	19	9	....	0.23	..	138	..
No. 5535	19	9	Mo 0.50	0.00	..	(88) 91	100
No. 5555	19	9	Mo 0.50	0.26	..	133	152
No. 5536	19	9	Mo 1.00	0.00	..	(98)	100
No. 5556	19	9	Mo 1.00	0.30	..	160	164
No. 5537	19	9	Mo 3.00	0.00	..	(90)	100
No. 5557	19	9	Mo 3.00	0.21	..	140	155
No. 5538	19	9	Cb 0.80	0.00	..	(103)	100
No. 5558	19	9	Cb 0.80	0.22	..	131	127
No. 5539	19	9	Ti 0.60	0.00	..	(122)	100
No. 5559	19	9	Ti 0.60	0.38	..	177	145
No. 5540	19	9	Cu 1.00	0.00	..	(89)	100
No. 5560	19	9	Cu 1.00	0.25	..	151	169
No. 5541	19	9	C 0.15	0.00	..	(88)	100
No. 5561	19	9	C 0.15	0.24	..	127	145
No. 5542	19	9	C 0.25	0.00	..	(89)	100
No. 5562	19	9	C 0.25	0.21	..	68	76
No. 5543	25	12	....	0.00	..	(74)	100
No. 5563	25	12	....	0.25	..	150	204
No. 5544	25	12	Mo 3.00	0.00	..	(85) 87	100
No. 5564	25	12	Mo 3.00	0.26	..	141	165
No. 5545	28 to 30	8 to 10	....	0.00	..	(120)	100
No. 5565	28 to 30	8 to 10	....	0.18	..	170	142
No. 5546 (As-cast)	18	1.0 max.	....	0.00	..	(116)	100
No. 5556 (As-cast)	18	1.0 max.	....	0.24	..	160	138
No. 5547 (As-cast)	13	0.5 max.	C 0.09 to 0.12	0.00	..	(39)	100
No. 5574 (As-cast)	13	0.5 max.	C 0.09 to 0.12	0.21	..	61	155
No. 5548	15	35	C 0.20	0.00	..	(127)	100
No. 5568	15	35	C 0.20	0.61	..	142	112
No. 5549	19	6	Mn 5.00	0.00	..	(104)	100
No. 5569	19	6	Mn 5.00	0.22	..	155	149
No. 5550	19	9	Mn 2.00	0.00	..	(102)	100
No. 5570	19	9	Mn 2.00	0.27	..	152	155
No. 5551	19	9	Si 2.50	0.00	..	(94)	100
No. 5571	19	9	Si 2.50	0.21	..	119	127

TABLE I.—*Continued*

Heat	Intended Composition, per cent[a]			Actual Bismuth Content, per cent	Drill Index	Saw Index[b]	
	Chromium	Nickel	Other			Compared to 19 Cr, 9 Ni or 25 Cr, 12 Ni	Compared to Materials of Same Analysis Without Bi
			WROUGHT ALLOYS				
No. 5278	18	8	C 0.06, Si 0.35, Mn 0.50	0.00	100	....	100
No. 5279	18	8	C 0.06, Si 0.35, Mn 0.50	0.31	194	....	134
No. 5280	12	..	C 0.06, Si 0.30, Mn 0.50	0.00	100	....	100
No. 5281	12	..	C 0.06, Si 0.30, Mn 0.50	0.10	..	....	172
No. 5288	18	8	C 0.06, Si 0.35, Mn 0.55	0.00	100	....	100
No. 5289	18	8	C 0.06, Si 0.35, Mn 0.55	0.25	119	....	143
No. 5294	18	8	C 0.06, Si 0.35, Mn 0.55	0.02	100	....	100
No. 5295	18	8	C 0.06, Si 0.35, Mn 0.55	0.31	160	....	145

[a] Carbon, 0.08 to 0.10 per cent; silicon, 1.00; manganese, 0.80; phosphorus, 0.04, max.; sulfur, 0.03, max. Actual analyses of scattered heats show the compositions to be very close to those intended.

[b] Saw index numbers in parentheses are calculated values; all other values are actual. The calculated values for the materials compared to 19 Cr, 9 Ni or 25 Cr, 12 Ni alloys were obtained by dividing the index of the Bi containing alloy by the index obtained when compared to materials of same analysis and multiplying this value by 100.

0.1 per cent rouge in water on the specimens. An abrasive is necessary to insure reproducible results. In service, it is usually found that grit is present and initiates most galling. Load is then applied in set stages, until galling occurs or the load or torque limit of the machine is reached. The materials tested and the results of these galling tests are contained in Table IV. These data indicate an increase of approximately 100 per cent in resistance of the bismuth-containing alloys under the testing conditions described.

Weldability:

One-inch wrought bars of 19-9 and 25-12 containing bismuth were swaged at approximately 1800 F. to $\frac{3}{16}$-in. rods. Using these rods as bare metal electrodes in an arc welder, successive layers of weld metal were laid on the ends of bars of the same stock from which the rods were swaged. The weld metal was approximately $\frac{3}{4}$ in. in thickness. The remainder of the swaged rod was commercially coated. The same procedure as described above for the bare rods was applied to these coated rods. To determine bismuth losses on weld metal deposited from both the bare and coated rods, samples for analysis were obtained of the weld metal, weld-base metal and weld metal near the base.

TABLE II.—CHARPY IMPACT TEST RESULTS.

Heat	Composition, per cent			Impact, ft-lb.		
	Chromium	Nickel	Bismuth	Low	High	Average of 4
No. 5552	19	9	None	61	75	66
No. 5553	19	9	0.23	48½	61	56
No. 5543	25	12	None	61	71½	65
No. 5563	25	12	0.21	43	49½	47

Table V contains the analytical data on the bismuth content after arc welding. It is obvious from the data in the table that the bismuth content of the weld metal, weld-base metal interface, and weld metal near the interface has been decreased by arc welding. The bismuth content of 0.06 per cent remaining in the weld metal would indicate a saw index of approximately 110 to 115 which represents some increase in machinability. During the arc-welding operation

TABLE III.—RESULTS OF SHORT-TIME TENSION TESTS AT ROOM TEMPERATURE AND 1800 F. ON 19 PER CENT CHROMIUM, 9 PER CENT NICKEL A.C.R.I. TEST BLOCK SPECIMENS.

All Specimens water quenched from 2075 F.

Heat	Bismuth Content, per cent	0.1 per cent Elongation, psi.	0.2 per cent Elongation, psi.	Proportional Limit, psi.	Tensile Strength, psi.	Elongation, per cent	Reduction of Area, per cent[a]
ROOM TEMPERATURE							
No. 5455-1A	0.0	31 000	32 800	23 000	73 600	65.5	70.4
No. 5455-1C	0.0	31 900	33 700	23 000	76 800	59.0	68.8
Average		31 450	33 250	23 000	75 200	62.3	69.6
No. 5455-2A	0.20	31 600	33 300	22 000	74 500	59.5	73.0
No. 5455-2C	0.20	31 400	32 900	22 000	72 500	58.5	71.6
Average		31 500	33 100	22 000	73 500	59.0	72.3
No. 5455-3A	0.26	28 300	30 300	19 000	68 900	58.0	75.1
No. 5455-3C	0.26	28 600	30 400	20 000	69 700	57.0	73.0
Average		28 450	30 350	19 500	69 300	57.5	74.1
1800 F.							
No. 5455-1B	0.0	4200	4500	3000	6800	53.5	57.5
No. 5455-1D	0.0	4350	4700	3500	6800	47.5	59.8
Average		4300	4600	3250	6800	50.5	58.7
No. 5455-2B	0.20	4700	5050	3000	6700	17.0	18.1
No. 5455-2D	0.20	4350	4700	3000	6650	14.0	28.2
Average		4550	4900	3000	6700	15.5	23.2
No. 5455-3B	0.26	4500	4800	3000	6650	8.5	13.4
No. 5455-3D	0.26	4050	4300	3500	6250	6.0	10.4
Average		4300	4550	3250	6450	7.5	11.9

[a] Approximate value only.

TABLE IV.—RESULTS OF GALLING TESTS ON STAINLESS STEELS.

Heat	Composition, per cent	Speed, rpm.	Galling Load, kg.			
			Test No. 1	Test No. 2	Test No. 3	Test No. 4
No. 5552	19 Cr, 9 Ni	213	80	80	..	..
No. 5388	19 Cr, 9 Ni	213	..	..	100	80
No. 5553	19 Cr, 9 Ni, 0.23 Bi	213	over 100[a]	over 100	..	..
No. 5395	19 Cr, 9 Ni, 0.32 Bi	213	..	..	over 190	over 140
No. 5552	19 Cr, 9 Ni	426	0[b]	0	..	..
No. 5553	19 Cr, 9 Ni, 0.23 Bi	426	15	15	..	..
No. 5395	19 Cr, 9 Ni, 0.32 Bi	426	..	..	..	25
No. 5543	25 Cr, 12 Ni	213	80	80	..	..
No. 5389	25 Cr, 12 Ni	213	..	..	100	..
No. 5563	25 Cr, 12 Ni, 0.30 Bi	213	over 100	over 100	..	..
No. 5398	25 Cr, 12 Ni, 0.34 Bi	213	..	..	over 140	over 200
No. 5543	25 Cr, 12 Ni	426	0	0	..	..
No. 5563	25 Cr, 12 Ni, 0.30 Bi	426	5	5	..	..
No. 5398	25 Cr, 12 Ni, 0.34 Bi	426	..	..	15	20

[a] A load shown as over a specified value indicates that galling was not obtained in the test.
[b] A zero value indicates that galling occurred at an unmeasurable value of 1 kg. or less.

TABLE V.—ANALYTICAL DATA ON THE RECOVERY OF BISMUTH IN THE BASE AND WELD METAL AFTER ARC WELDING WITH BARE AND COVERED ELECTRODES.

Original Composition of Base and Weld Metal, per cent	Bismuth Content Using Bare Electrodes, per cent			Bismuth Content Using Covered Electrodes, per cent		
	Weld Metal	Base Metal plus Weld Metal	Weld Metal Near Weld Interface	Weld Metal	Base Metal plus Weld Metal	Weld Metal Near Weld Interface
19 Cr, 9 Ni, 0.16 Bi[a]	0.04	0.11	0.05	0.06	0.18	0.05
25 Cr, 12 Ni, 0.29 Bi[b]	0.06	0.14	0.06	0.06	0.24	0.06

[a] C 0.10, Mn 0.56, Si 0.65.
[b] C 0.06, Mn 0.79, Si 0.86.

there appeared to be greater evolution of gas when using the bismuth bearing rods. The weld deposits resulting from the use of coated rods, however, were sound and have been shown to be somewhat superior in corrosion resistance to the casting (of the same composition) on which they were deposited.

TABLE VI.—EFFECT OF BISMUTH ON THE CORROSION RESISTANCE IN SEVERAL ACID MEDIA.
Specimens 4¼ in. by ¼ in. by ⅛ in., surface ground on all faces.

Heat	Alloy Type	Actual Bismuth Content, per cent	Heat Treatment[a]	Increase in Electrical Resistance, per cent[c]										
				Copper Sulfate[d]				Nitric Acid[e]				Nitric-Hydrofluoric[f]		
				24 hr.	90 hr.	163 hr.	284 hr.	72 hr.	72 hr.	144 hr.	144 hr.	96 hr.	192 hr.	192 hr.
				Measured				Calc'd.	Meas'd.	Calc'd.	Meas'd.	Meas'd.	Calc'd.	Meas'd.
No. 6390	19 Cr, 9 Ni, 0.08 C	0	S	320	1400	22 100	..	4.9	50.0	16.4	254	171	34	1755
No. 6396		0.26	S	7	34	110	900	1.3	10.9	4.7	51	48	31	136
No. 6390		0	WQ	0	0	0	0	0.8	0.7	2.4	2.5	1.4	2.6	3.2
No. 6396		0.26	WQ	0	0	0	0	0.3	0.3	0.9	1.4	0.0	2.2	3.1
No. 6391	19 Cr, 9 Ni, 0.08 C + 0.8 Cb	0	S	0	0	0	0	0.3	0.0	0.7	1.8	10.0	24	33
No. 6397		0.19	S	0	0	0	0	0.3	0.3	0.6	0.7	7.4	21.9	26.9
No. 6391		0	WQ	0	0	0	0	0.2	0.0	0.4	0.0	0.5	0.9	1.4
No. 6397		0.19	WQ	0	0	0	0	0.4	0.3	0.9	1.0	0.3	0.7	1.5
No. 6392	19 Cr, 9 Ni, 0.08 C + 0.6 Ti	0	S	0	0	0	0	0.5	0.7	2.0	3.5	5.8	8.4	12.8
No. 6398		0.21	S	0	0	0	0	1.0	2.1	3.2	6.8	13,1	26.0	32.2
No. 6392		0	WQ	0	0	0	0	0.3	0.2	0.8	0.7	0.9	1.1	2.1
No. 6398		0.21	WQ	0	0	0	0	0.4	0.3	1.0	1.4	0.3	1.2	1.1
No. 6393[b]	19 Cr, 9 Ni, 0.08 C + 3.0 Mo	0	S	0	0	0	0	13.9	24.4	93.5	136	17.4	36.5	41.0
No. 6399[b]		0.19	S	0	0	0	0	16.2	28.1	124	161	16.2	37.3	45.1
No. 6393[b]		0	WQ	0	0	0	0	1.4	1.3	8.2	11.0	0.1	0.5	0.5
No. 6399[b]		0.19	WQ	0	0	0	0	1.1	1.6	11.0	13.8	0.5	0.4	1.0

[a] S = sensitized for 100 hr. at 1200 F. after quench annealing. The titanium heats were given a 3 hr. stabilizing treatment at 1575 F. before sensitizing.
WQ = Water quenched from 2050 F.

[b] The first period in nitric acid was 53 instead of 72 hr.

[c] Meas'd. = Measured increase in electrical resistance; Calc'd. = calculated increase in electrical resistance from weight loss data.

[d] 47 ml. H_2SO_4 + 13 g. $CuSo_4 \cdot 5H_2O$ per liter at boiling.

[e] Boiling 65 per cent nitric acid solution.

[f] Solution of nitric acid (Sp.g. 1.42) 20 per cent by volume and hydrofluoric acid (48 per cent) 4 per cent by volume at 140 F.

Corrosion Resistance

With these indications that mechanical properties and weldability of the corrosion-resisting casting alloys are not impaired to any important degree, attention was turned to evaluation of the effect of bismuth upon corrosion resistance in general.

Alloys of 19-9 and 25-12 with and without 3 per cent molybdenum, each of these without bismuth and with about 0.25 per cent bismuth were subjected to the action of 5 per cent $FeCl_3$ + 10 per cent NaCl for 24 hr. and examined for pitting. No effect of bismuth in increasing or reducing pitting appeared. The beneficial effect of molybdenum was not impaired by the presence of bismuth.

Specimens of a number of the alloys with and without bismuth were studied as to intergranular attack in three test solutions, namely copper sulfate - sulfuric acid, nitric acid, and nitric-hydrofluoric acids. Intergranular penetration was measured by the percentage change in the electrical resistance of specimens of uniform dimensions and general attack was measured by weight loss. The latter figures were translated to a calculated percentage increase in electrical resistance. No effect of bismuth was shown in any case save that of sensitized 19-9. A few of the voluminous data on which this statement is based are shown in Table VI. In the case of the sensitized 19-9, bismuth had the effect of retarding the rate of intergranular attack, as the table shows.

The resistance of the alloys to general attack in a number of acid media was determined. A few representative data from these tests are presented in Table VII.

TABLE VII.—CORROSION DATA FOR SELECTED ALLOYS.

Corrodent	Weight Loss, mg. per sq. in. per day											
	Heat No. 5552C	Heat No. 5553	Heat No. 5543	Heat No. 5563	Heat No. 5537	Heat No. 5557	Heat No. 5544	Heat No. 5564	Heat No. 5538	Heat No. 5558	Heat No. 5539	Heat No. 5559
	19 Cr-9 Ni	19 Cr-9 Ni-0.23 Bi	25 Cr-12 Ni	25 Cr-12 Ni-0.25 Bi	19 Cr-9 Ni-3 Mo	19 Cr-9 Ni-3 Mo-0.21 Bi	25 Cr-12 Ni-3 Mo	25 Cr-12 Ni-3 Mo-0.26 Bi	19 Cr-9 Ni-0.80 Cb	19 Cr-9 Ni-0.80 Cb-0.22 Bi	19 Cr-9 Ni-0.60 Ti	19 Cr-9 Ni-0.60 Ti-0.38 Bi
½% HCl—at 35 C. for 72 hr.	30.0	3.3	....	....	....	....	....	....	77	5.0	86	1
1% HCl—at 35 C. for 72 hr.	75.0	85.4	207	62.8	7.9	3.4	....	....	60	134	126	1.2
5% HCl—at 35 C. for 72 hr.	74.0	104.0	544.0	745.0	135.5	180.5	53.9	0.5	227.0	865.5	492.0	300.0
8% HCl—at 35 C. for 72 hr.	....	....	....	....	....	....	506	451	....	....	....	....
10% HCl—at 35 C. for 72 hr.	....	....	1435.0	1741.0	653.0	793.0	903.0	938.0	....	....	....	....
3% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	....	....	....	....	125	1.5	....	....
5% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	....	....	....	....	137	208	....	....
7% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	....	....	....	....	....	....	180	1.4
7.5% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	....	....	....	....	187	296	201	29
10% H_2SO_4—at 35 C. for 72 hr.	68.9	94.0	735.0	0.70	0.1	0.1	0	0	157.2	344.0	383.0	10.9
13.5% H_2SO_4—at 35 C. for 72 hr.	....	....	973	0	....	....	....	....	....	....	....	....
15% H_2SO_4—at 35 C. for 72 hr.	....	....	1290	1170	....	....	....	....	....	....	503	94
20% H_2SO_4—at 35 C. for 72 hr.	....	....	1400	1460	0.20	0.10	0.8	0.9	....	....	....	....
30% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	1	3	2	1	....	....	....	....
40% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	1	1	0.2	0.1	....	....	....	....
50% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	3	2	0.2	0.1	....	....	....	....
55% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	207	99	0.1	1.0	....	....	....	....
70% H_2SO_4—at 35 C. for 72 hr.	....	....	....	....	....	....	1.0	1.0	....	....	....	....
60% H_3PO_4—at 80 C. for 72 hr.	0.06	0.22	....	....	....	....	....	....	....	....	....	....
70% H_3PO_4—at 80 C. for 72 hr.	0.01	0.21	0.10	0.04	0.12	0.04	0.31	0.09	....	....	....	....
85% H_3PO_4—at 80 C. for 72 hr.	0.16	0.18	0	0	0.04	0.00	0	0	....	....	....	....
65% HNO_3—at boiling:												
First 48-hr. period	4.7	5.7	2.2	2.3	3.3	5.1	3.9	3.3	4.3	3.5	6.2	7.8
Second 48-hr. period	2.7	4.0	1.5	1.8	5.5	5.4	3.3	3.0	3.3	2.8	15.4	8.5
Glacial acetic at boiling for 48 hr.	2.1	0.9	0.03	0.18	....	....	0.6	0.05	....	....	....	....
10% Lactic acid at 65 C. for 72 hr.	0.95	0.04	....	....	....	....	....	....	....	....	....	....
20% Lactic acid at 65 C. for 72 hr.	956	0.14	....	....	....	....	....	....	....	....	....	....
30% Lactic acid at 65 C. for 72 hr.	743	1.26	....	....	....	....	....	....	....	....	....	....
50% Lactic acid at 65 C. for 72 hr.	148	193	0.29	0.32	0.09	0.02	0.03	0.02	....	....	....	....
5% Lactic acid at boiling for 48 hr.	....	....	79.2	0.10	....	....	....	....	....	....	....	....
10% Lactic acid at boiling for 48 hr.	422	372	46.2	128	0.10	0.20	....	....	....	....	....	....
Dilute mixed acids:[a]												
First period of 2 hr.	335	638	12.1	1.6	4.7	3.9	....	....	....	....	....	....
Second period of 2 hr.	890	4.1	327	0	0	0	....	....	....	....	....	....
Third period of 4 hr.	9.4	0.5	0.1	0	0.4	0.1	....	....	....	....	....	....
Fourth period of 4 hr.	2.0	0	0.4	0	0	0	....	....	....	....	....	....
Fifth period of 8 hr.	3.7	0.6	0	0	0	0	....	....	....	....	....	....
Sixth period of 8 hr.	0.4	0	0	0	0	0	....	....	....	....	....	....

[a] Boiling solution containing 1 part HNO_3 + 3 parts H_2SO_4 diluted to 0.5 per cent acid figured as H_2SO_4.

In general the addition of bismuth decreases the corrosion resistance when conditions are such that the alloys are in the active state of corrosion. This is least important. When conditions are such that the alloys are in the passive state of corrosion, the addition of bismuth either has no appreciable effect or, in some instances, increases the range of passivity. Since many of the corrosion tests listed in the table were conducted under borderline conditions of passivity and activity, the results of some of them are not entirely consistent.

Galvanic behavior of couples of bismuth-containing and bismuth-free alloys was studied in various concentrations of H_2SO_4. No evidence was found of detrimental effect of bismuth on the corrosion of such couples.

Salt-spray tests carried on for 4200 hr. on 19-9 and 25-12 with and without 3 per cent molybdenum, each with and without about 0.25 per cent bismuth showed no effect due to bismuth. Sea-water exposure tests are under way. The specimens will be examined before the June meeting and comment then made on the results.

Addition of Bismuth to the Alloys

The experimental heats were made in high frequency induction furnaces. The bismuth was added to the majority of the bismuth bearing steels as a master alloy of nickel bismuth containing 70 per cent bismuth and 30 per cent nickel. To some heats it was added as lumps of the granular form of the metal, as sodium bismuthate, as bismuth trioxide and as a master alloy of manganese-bismuth (80 Bi - 20 Mn). The master alloys Ni-Bi and Mn-Bi had relatively high melting points, and were prepared by simply melting the nickel or manganese in an open crucible of the high-frequency type furnace and then adding the proper amount of bismuth to the molten metal. Another method of preparation consisted of pouring molten nickel into a crucible containing the proper amount of bismuth. The recovery of bismuth in both methods of preparation of the master alloy was practically complete.

There was considerable effervescence when bismuth, regardless of the form, was added to the alloy steels. The boiling point of bismuth is about 300 F. below the temperature of the molten steel to which it was added. This low boiling point is undoubtedly the reason for the effervescence. All the bismuth steels fumed, but in no case was the effervescence violent if the bismuth was added in small quantities. Less fuming was noted with the furnace power on than when pouring the metal. Providing the induction furnace with a cover top or "capping" the heats was beneficial. Addition of bismuth as bismuth-nickel alloy minimizes fuming. When elementary bismuth is used as the addition, it should preferably be in the form of pea-size lumps.

The crucible used in melting the bismuth-containing alloys appeared to be in better condition than the crucible used in preparing the bismuth-free alloys after an equivalent number of heats had been melted in both.

Commercial test melts show that arc furnaces are suitable for the preparation of the bismuth-containing alloys.

Bismuth Recovery in the Alloys

The recovery of bismuth in the 19 Cr - 9 Ni and 25 Cr - 12 Ni is governed to some degree by the time interval from the addition of bismuth to the pouring of the metal, by the method used, and by the amount of the addition. Considering both foundry procedure and maximum recovery, the introduction of bismuth should be in the form of a 0.5 to 1 per cent addition of the master alloy Ni-Bi. The heat should be held 10 to 20 min. before pouring. A 0.5 per cent addition of the master alloy gave virtually the same bismuth content as a 1 per cent addition.

The recovery of bismuth in the majority of the heats melted and cast was approximately 0.20 to 0.30 per cent. The exceptions were heat No. 5568 (15 Cr - 35 Ni), in which a bismuth content of 0.61 per cent was obtained, and heat No. 5559 (0.60 Ti), which retained 0.38 per cent bismuth.

Virtually the same recovery was noted on large induction furnace heats as was obtained on the smaller experimental heats. In the case of the large heats, capping the furnace appeared to be important in securing the maximum bismuth content.

No loss of bismuth was noted after subjecting the heats to the annealing treatment consisting of water quenching from 2050 F.

A button of bismuth or bismuth alloy was usually observed in the bottom of the crucible when the last metal was poured. This suggests why the bismuth content is relatively independent of the amount added and indicates that mechanical or chemical agitation should improve the bismuth recovery. Bismuth recovery is probably also affected by size and depth of the melt, capping, and general foundry procedure.

METALLOGRAPHIC EFFECT OF PRESENCE OF BISMUTH

Bismuth is, at least in part, present as metallic particles which appear to be practically pure bismuth. These hardly seem plentiful enough to account for the improved machinability, and the presence of widely distributed, submicroscopic dispersion of bismuth is suspected, though nothing of this sort has been revealed at high magnifications. The situation appears somewhat analogous to the early history of leaded steels, where the machine shop found improved machinability, the chemical analysis showed lead, but metallographic examination failed to indicate its presence.

Tentative interpretation of metallographic evidence to date indicates that the bismuth addition tends to favor the formation of ferrite, and there is some indication of increased carbon solubility in the presence of bismuth. The metallographic data will be presented in detail in another paper.

CONCLUSIONS

Laboratory tests and foundry experience of A.C.R.I. members lead to the conclusions that:

Small bismuth additions to the cast corrosion-resistant chromium and chromium-nickel alloys with and without molybdenum, columbium, titanium, etc., materially improve their machinability, affect the mechanical properties important in corrosion-resistant service in no important degree, improve the galling resistance, do not injure weldability, and in general exert no deleterious effect upon corrosion resistance to any corrodent so far studied. In certain specific cases, corrosion resistance is somewhat improved.

The advantage of improved machinability is gained without sacrifice of the important properties of these corrosion-resistant castings. A similar condition probably holds for the wrought alloys, but these have not yet been exhaustively studied. The hot working of bismuth-containing 19-9 and 25-12 alloys, while feasible, must be carried out at somewhat lower than usual temperatures.

The decreased ductility in short-time tension tests at 1800 F. indicates that care will be needed as to the type of applications to high-temperature service, but the maintained strength indicates that the load-carrying ability may be satisfactory. Because the pressing need for machinability is greater in the case of assemblies for corrosion resistance than in that of heat resistance, work to date has been concentrated on the former.

Acknowledgments:

The authors wish to express their thanks to H. W. Gillett and H. W. Russell of Battelle Memorial Institute for their interest and suggestions; to Russell Dayton and David I. Sinizer for a part of the experimental work; to Clyde E. Williams, Director, Battelle Memorial Institute, and the Technical Committee of the Alloy Casting Institute, for permission to publish.

DISCUSSION

Mr. F. L. LaQue[1] (*presented in written form*).—The authors mentioned that specimens of cast stainless steels with and without the addition of about 0.25 per cent bismuth were being tested in sea water. These tests, which were under the direction of the writer, were completed in May, 1941, and represented an exposure test period of about 8 months.

TABLE I.—RESULTS OF EXPOSURE OF CAST STAINLESS STEELS CONTAINING BISMUTH IN FLOWING SEA WATER FOR EIGHT MONTHS. DIMENSIONS OF SPECIMENS 6 BY 12 BY ½ IN.

Material	Weight Loss of Specimen, g.		
	Spec. 1	Spec. 2	Average
19 Cr 9 Ni	54	34	44
19 Cr 9 Ni, 0.25 Bi	38	32	35
25 Cr 12 Ni, 3 Mo	3	17	10
25 Cr 12 Ni, 3 Mo, 0.25 Bi	14	4	9

Material	Pitting Data					
	Maximum Depth of Pitting, mils			Average of Ten Deepest Pits, mils		
	Spec. 1	Spec. 2	Avg.	Spec. 1	Spec. 2	Avg.
19 Cr 9 Ni	303	373	338	190	233	212
19 Cr 9 Ni, 0.25 Bi	300	380	340	195	243	219
25 Cr 12 Ni, 3 Mo	253	82	168	95[a]	49[a]	72
25 Cr 12 Ni, 3 Mo, 0.25 Bi	125	230	178	66[b]	195[c]	130

[a] 8 pits.
[b] 7 pits.
[c] 2 pits.

The numerical results of this test are given in the accompanying Table I. They are in line with the conclusion reached by the authors with respect to other corrosive media that the addition of bismuth to the alloys studied had no significant effect on their resistance to attack by natural sea water.

The specimens used in this test were in the form of cast plates which measured 6 by 12 by ½ in. These were furnished with one side left as cast and the other machined; in addition, one-half of the machined side was surface ground. The number of pits which had started from the machined surfaces was greater than from the as-cast surfaces, but there was little difference in the depth of the deepest pits from the two types of surface.

The specimens were bolted to a monel rack from which they were insulated by means of bakelite sleeves and washers and immersed in flowing sea water at an average depth of about 2½ ft. at the plant of the Ethyl Dow Chemical Co., Wilmington, N. C. The average velocity of flow was about 1 ft. per sec. However, the specimens acquired thick deposits of marine organisms which tended in time to reduce the true velocity of the water across the surfaces of the alloys.

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[1] Metallurgist, Development and Research Division, International Nickel Co., Inc., New York, N. Y.

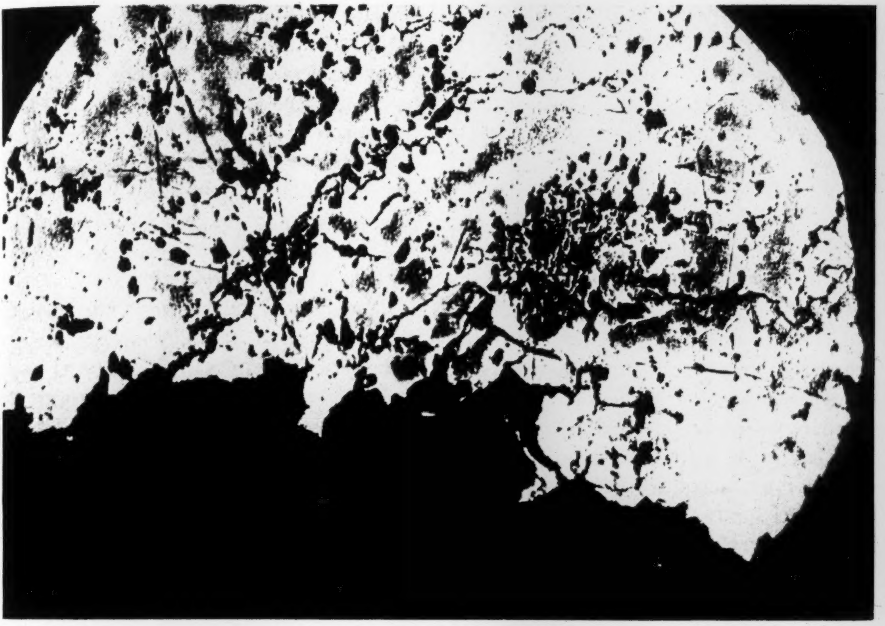

FIG. 1.

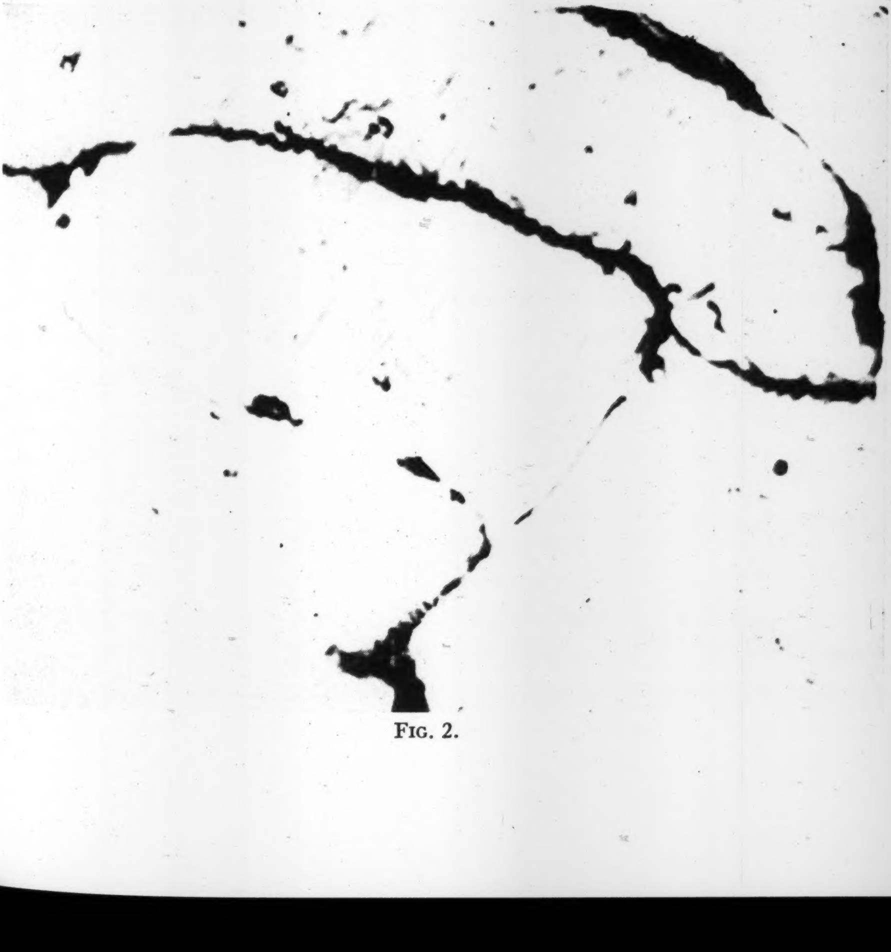

FIG. 2.

There was no evidence that the presence of bismuth in the alloys had any effect in suppressing the adherence or growth of the marine organisms. The season of exposure was from September to May during which the temperature of the water varied from about 10 C. to 25 C. (50 F. to 77 F.).

Corrosion took the form of rather large and occasionally deep pits, many of which appeared definitely to be associated with subsurface porosity or actual voids which became enlarged as a result of concentration cell effects after the initial corrosion penetration of either the casting skin or the machined surface. However, as mentioned previously, neither the general appearance of the specimens nor the results of the weight-loss determinations and pit-depth measurements indicated that the presence of bismuth had any significant effect on the incidence or progress of corrosion of these specimens.

MR. F. B. FOLEY.[2]—Confirming the statements of the authors regarding the hot rolling of these alloys, we find that the addition of bismuth has the effect of lowering the temperature at which hot working may be successfully accomplished to not over 1900 F. Machinability tests run in a manner similar to that described in the paper showed wrought bismuth-bearing 18 per cent chromium, 8 per cent nickel alloy could be sawed as readily as selenium-bearing 18 per cent chromium, 8 per cent nickel and could be drilled much more easily.

MESSRS. E. K. SMITH[3] AND H. C. AUFDERHAAR[4] (*by letter*).—It is noted on page 9 that the authors come to the conclusion that bismuth is at least in part present as metallic particles of practically pure bismuth. The authors suspect submicroscopic dispersions of bismuth, although they found no evidence of this under high magnifications.

It is possible that the work done by the present writers along these lines may throw some additional information on microstructures.[5] In this case, the physical properties of the cast iron were lowered enormously, and machinability, of course, was greatly increased. Under the microscope it was seen that the grains of iron were more or less surrounded by a dark-colored material, which we supposed to be some oxide of bismuth. In the accompanying Fig. 1 it will be noted that the fracture of iron has followed these grains. Obviously, the intergranular material has been very weak, which accounts for the loss in strength of the cast iron. I believe that the photomicrograph was at 100 diameters unetched.

The accompanying Fig. 2 shows one of the grains at 3500 diameters. It will be noted that the grain is almost completely surrounded by some dark material, which we believed to be bismuth oxide. It will be interesting to note whether in their continued investigation the authors find any evidence of such precipitation of oxides around the grain borders.

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[3] Metallurgist, Electro Metallurgical Co., Detroit, Mich.

[4] Metallurgist, Electro Metallurgical Co., Chicago, Ill.

[5] E. K. Smith and H. C. Aufderhaar, "Effect of Adding Bismuth to Cast Iron," *The Iron Age*, Vol. 128, July 9, 1931, p. 96 -100.

THE EFFECT OF SHOT BLASTING AND ITS BEARING ON FATIGUE

BY J. M. LESSELLS[1] AND W. M. MURRAY[1]

The term shot blasting is applied to the process whereby the outside surface of a metal is subjected to the impingement of iron shot. The effect of the chilled cast iron shot cold works the outer surface and also develops residual compressive stresses in this region. The result is that under controlled conditions considerable increase in fatigue life can be obtained. Subject to the reservations made in the paper this increase is obtained regardless of whether the original surface was machined.

This fact is of considerable economic importance because it is generally agreed that unmachined forgings[2] suffer considerable decrease in endurance limit due to the unmachined surface. This decrease increases with tensile strength. Shot blasting makes unmachined surfaces stronger in fatigue. As regards machined surfaces it is well known that the highest fatigue strength is associated with the highest degree of surface finish. Shot blasting of machined surfaces imparts an increase in fatigue strength without recourse to high degree of polishing. As stated above these benefits have a distinct economic value.

The shot-blasting process was possibly suggested by the work of Herbert[3] on "cloudburst" hardening which was followed by that of Frye.[4] The advantages of shot blasting had however been appreciated for 10 yr. prior to this latter publication. Zimmerli[5] has also made valuable contributions to the subject. The process is now widely used by certain industries and its application is continually extending. Its present state of development must be associated with the names of J. O. Almen, M. Olley, F. P. Zimmerli, W. H. Wallace and H. H. Clark.

This paper describes some experimental work which has been done on different steels at the Massachusetts Institute of Technology and discusses the results obtained. It is hoped that the paper will arouse considerable discussion so that a better understanding of this important process may be the result.

METHOD OF SHOT BLASTING

Considerable data have been published on the technique of the process.[5] There are two methods in use for imparting velocity to the shot. The older method is by use of air under a pressure of 25 psi. and a more recent method by the use of a wheel revolving at high velocity into which the shot is fed. The tests described in this paper, except those for

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[1] Associate Professor of Mechanical Engineering, and Assistant Professor of Mechanical Engineering, respectively, Massachusetts Institute of Technology, Cambridge, Mass.

[2] G. A. Hankins, M. L. Becker and H. R. Mills, "Further Experiments on the Effect of Surface Conditions on the Fatigue Resistance of Steels," *Journal*, Iron and Steel Inst., May, 1936 No. 1, p. 399.

[3] E. G. Herbert, "Work Hardening of Steel by Abrasion," *Engineering*, p. 470 (1927).

[4] J. H. Frye and G. L. Kehl, "Fatigue Resistance of Steel as Affected by Some Cleaning Methods," *Transactions*, Am. Soc. Metals, p. 192 (1938).

[5] F. P. Zimmerli, "Shot Blasting and Its Effects on Fatigue Life," *Preprint No. 51*, Am. Soc. Metals (1940).

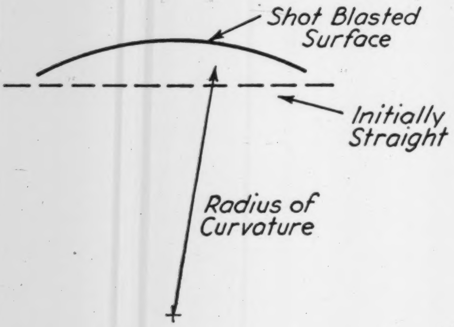


FIG. 1.—Strip for Determining the Stress Produced by Shot Blasting.

S.A.E. 1045, were made on material shot blasted by the latter process. The S.A.E. 1045 was shot blasted by air pressure.

The size of the shot is important and is generally limited to 0.025 in. to 0.030 in. Zimmerli mentions shot $\frac{1}{64}$ in. and $\frac{3}{64}$ in. in diameter. The Eaton Manufacturing Co. uses shot which falls within the specification of 50 per cent between 0.032 in. and 0.023 in. in diameter and 50 per cent between 0.023 in. and 0.016 in. in diameter. A typical analysis is:

Total Carbon, per cent	Combined Carbon, per cent	Graphitic Carbon, per cent	Silicon, per cent	Manganese, per cent	Sulfur, per cent	Phosphorus, per cent
3.45	3.30	0.15	1.80	0.55	0.15	0.46

EFFECT OF SHOT BLASTING

The effect of the shot is to cold work the outer region of the metal and induce considerable compressive stresses in these regions.

In such a process it is of interest to determine the depth of the cold-worked surface. Two different approaches have been made. Almen used a steel strip $\frac{3}{4}$ in. by 0.045 in. thick held in a suitable jig while being shot blasted on the outer surface. On removal from the jig the strip assumed a form indicated in Fig. 1.

By removing material from the shot-blasted surface in successive steps and measuring the (ρ) radius of curvature in inches at each stage a curve could be plotted as shown in Fig. 2.

After approximately 0.006 in. had been removed the strip assumed a straight form. This varied with the hardness of the material of which the strip was made, but the above depth is representative.

A further approach by use of X-ray diffraction methods was made by J. T. Norton of the Massachusetts Institute of Technology on a shot-blasted section from a 1-in. diameter fatigue test bar, S.A.E. X4340 steel, supplied by the authors. The different patterns ob-

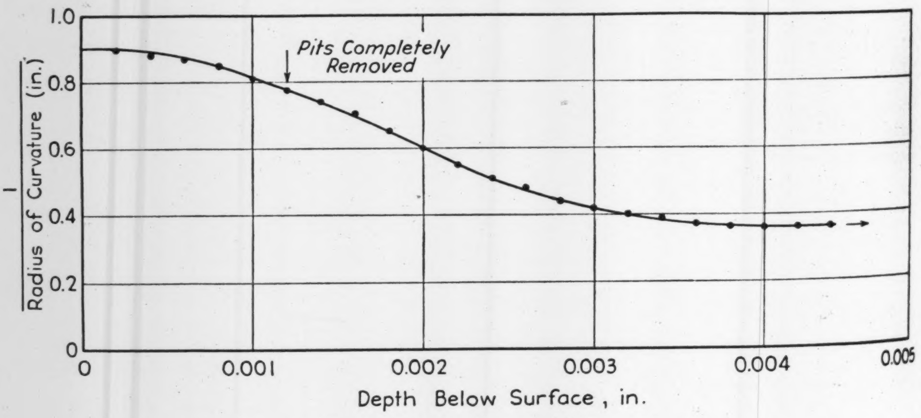
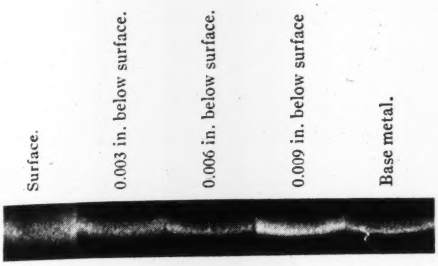


FIG. 2.—Curve Showing Depth of Shot Blasting.


Surface.
0.003 in. below surface.
0.006 in. below surface.
0.009 in. below surface
Base metal.

Diffraction patterns at different depths below surface.

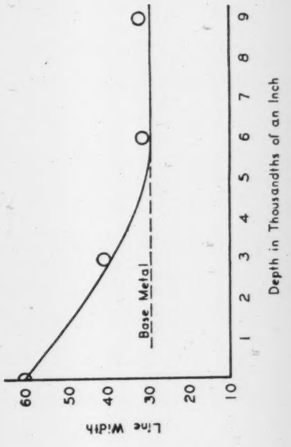


FIG. 4.—Band Width in X-ray Pattern.

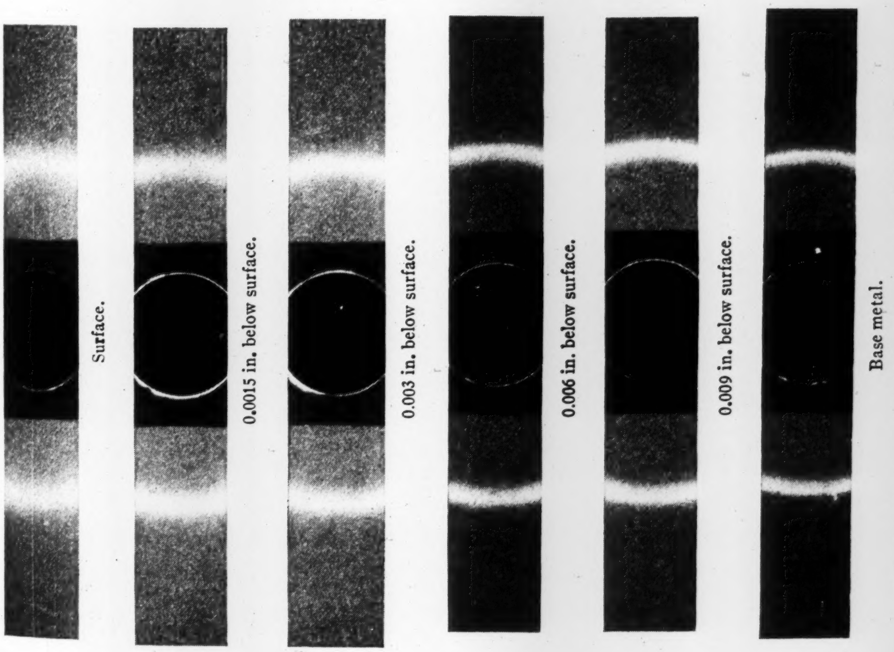

Surface.
0.0015 in. below surface.
0.003 in. below surface.
0.006 in. below surface.
0.009 in. below surface.
Base metal.

FIG. 3.—X-ray Diffraction Pattern After Shot Blasting.

tained are shown in Fig. 3 and a plot of band width is shown in Fig. 4.

This also confirmed Almen's work and it appears safe to conclude that the thickness of the cold-worked region is of the order of 0.006 in.

The type of stress distribution which prevails after shot blasting must therefore be as represented in Fig. 5.

SHOT BLASTING OF MACHINED SURFACES

Tension and fatigue tests were made on a wide variety of steels for the unshot-blasted and shot-blasted conditions which had been heat treated to different hardnesses and machined prior to shot blasting.

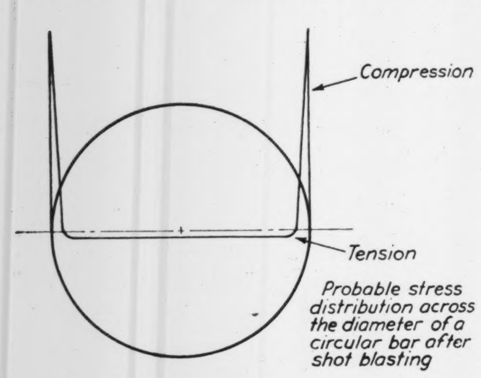

Compression
Tension
Probable stress distribution across the diameter of a circular bar after shot blasting

FIG. 5.—Probable Stress Distribution After Shot Blasting.

The chemical analyses of the steels investigated are given in Table I.

The Armco iron and S.A.E. 1045 were heat treated in bar form $\frac{1}{2}$ in. in diameter and $\frac{3}{4}$ in. in diameter, respectively, and machined and shot blasted. The S.A.E. 9260 was also heat treated in bar form $\frac{1}{2}$ in. in diameter and the S.A.E. X4340 2 in. in diameter.

The specimens of Armco iron, S.A.E. 9260, and S.A.E. X4340 were shot blasted and stress relieved for 20 min. at 400 F. The S.A.E. 1045 was given no stress relief after shot blasting.

The tension tests on the S.A.E. 1045

TABLE I.—ANALYSES OF STEELS.

Material	Carbon, per cent	Manganese, per cent	Phosphorus, per cent	Sulfur, per cent	Silicon, per cent	Nickel, per cent	Chromium, per cent	Molybdenum, per cent
S.A.E. 1045	0.48	0.72	0.011	0.025	0.17	...	...	...
Armco iron	0.045	0.04	0.005	0.043	0.02	...	...	...
S.A.E. 9260	0.60	0.80	0.014	0.023	1.95	...	...	...
S.A.E. X4340	0.44	0.67	0.012	0.008	0.20	1.74	0.78	0.26

Heat Treatments

S.A.E. 1045 (Annealed).—Heated to 1475 F. for 30 min. and furnace cooled.

S.A.E. 1045 (Heat Treated).—Water quenched from 1500 F. and drawn at 800 F.

Ingot Iron.—Heated to 1700 F. for 1 hr. and furnace cooled.

S.A.E. 9260.—Oil quenched from 1600 F. and drawn at 520 F. for 30 min.

S.A.E. X4340.—Oil quenched from 1500 F. and drawn at 1150 F.

TABLE II.—EFFECT OF SHOT BLASTING ON TENSILE PROPERTIES.

Surface Condition	Tensile Strength, psi.	Yield Point, psi.	Proportional Limit, psi.	Elongation, per cent[a]	Reduction of Area, per cent	Brinell Hardness Number
S.A.E. 1045 (ANNEALED)						
Polished	86 700 88 800	46 200[b] 50 000	40 000 47 500	27.5 29.5	47 47	160 165
Shot blasted	88 100 88 000	47 400[b] 48 000	37 500 43 700	30.5 29.5	47.5 47.5	168 165
S.A.E. 1045 (HEAT TREATED)						
Polished	152 700	117 100[b]	85 000	17	56	311
Shot blasted	135 800 143 800	103 500[b] 109 500[b]	60 000 70 000	20 21	60 60	285 285
S.A.E. 9260						
Polished	322 000 316 000	294 000[b] 294 000[b]	184 000 210 000	.. ..	7 14	548[c] 570[c]
Shot blasted	306 000 304 000	283 000[b] 281 000[b]	210 000 210 000	7 10	39 37	526[c] 530[c]
ARMCO IRON						
Polished	46 500 46 600	23 200 22 400	18 200 17 200	37 37	68 70	117[c] 125[c]
Shot blasted	46 400 46 700	23 900 22 600	13 900 15 800	30 34	66 68	121[c] 120[c]
S.A.E. X4340						
Polished	145 000	128 500	110 000	19.5	55	278
Shot blasted	145 000	127 500	112 000	19.5	56	277

[a] All elongations determined in a length equal to four times the diameter.

[b] 0.2 per cent set.

[c] Equivalent Brinell Numbers from Vickers Readings.

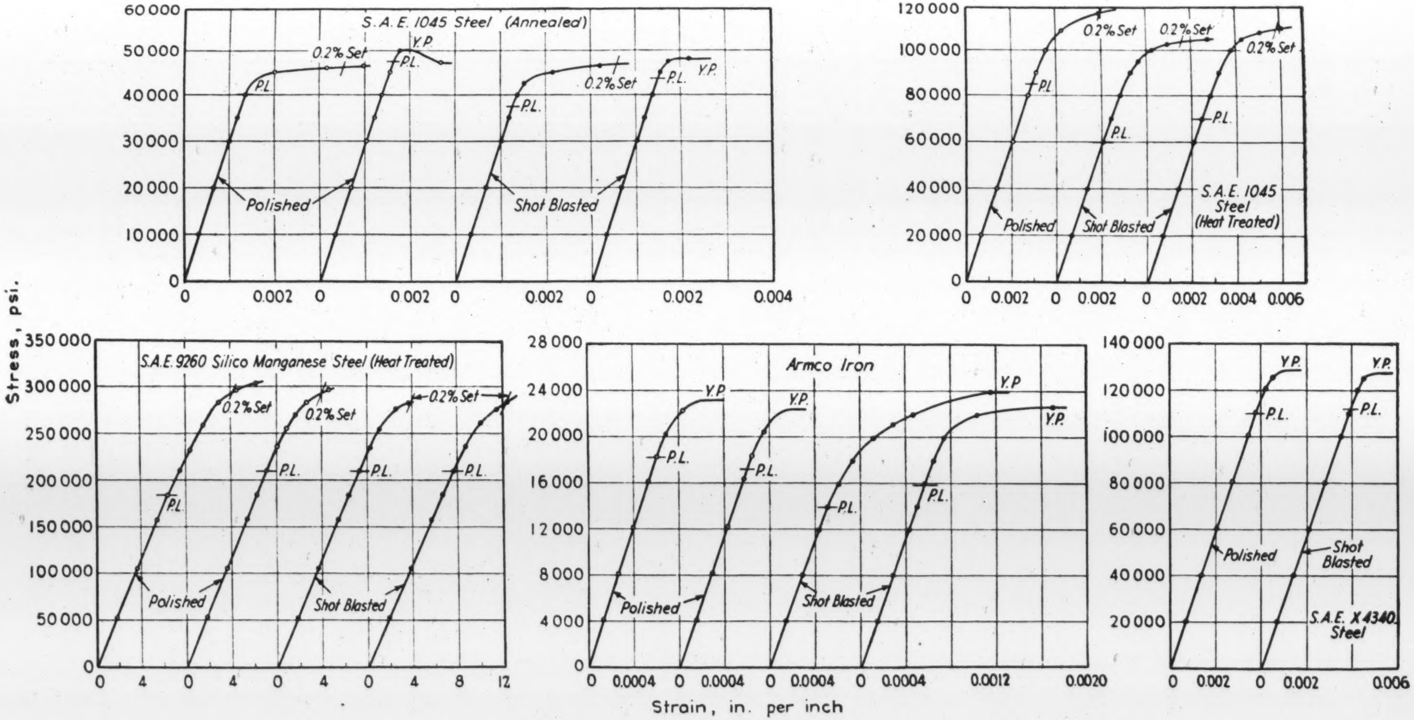


Fig. 6.—Tension Test Diagrams.

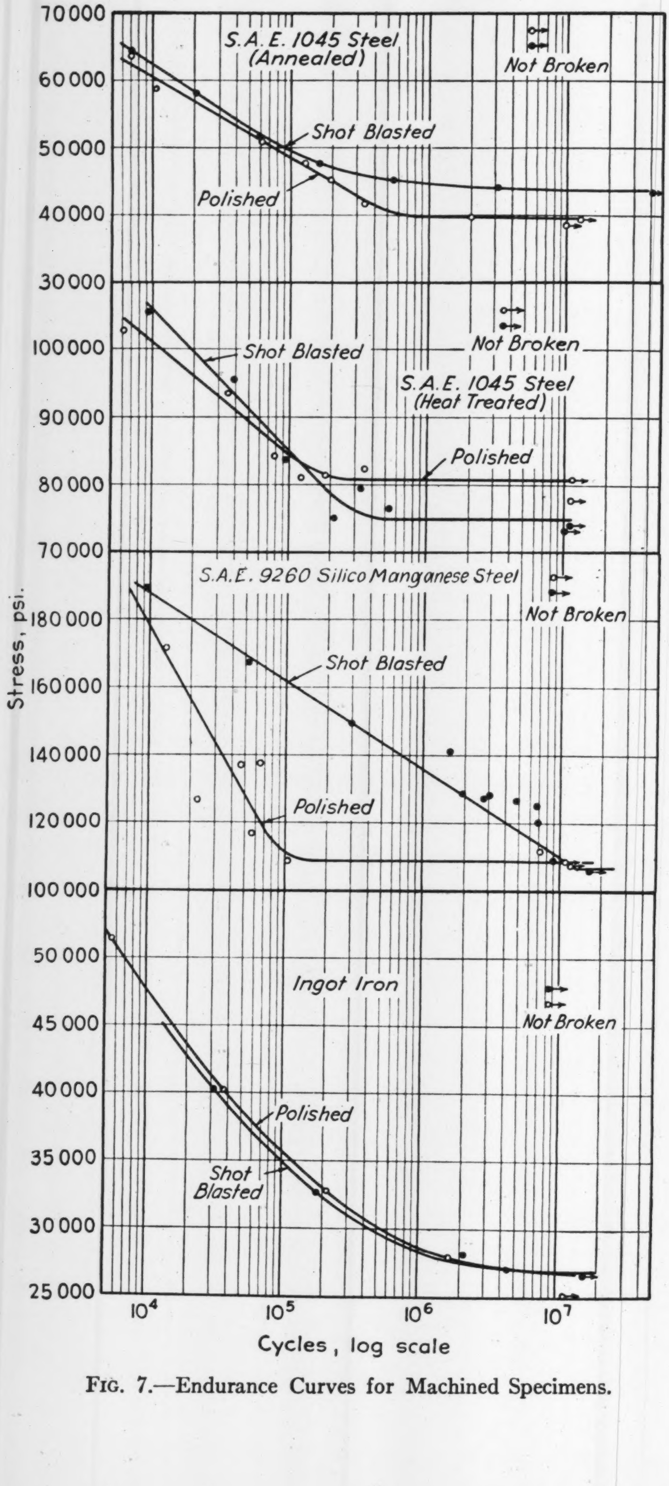


FIG. 7.—Endurance Curves for Machined Specimens.

and X4340 were made on standard 0.5 ± 0.005-in. diameter bars. For the S.A.E. 9620 and Armco iron smaller specimens were used. The fatigue tests, unless otherwise stated, were made on an R. R. Moore type machine operating at 1750 rpm. The minimum diameters of these specimens were 0.20 and 0.25 in.

The curves for the elastic stages of the tension tests were obtained by the use of Martens Mirror, Huggenberger, and de Forest-Ruge extensometers. These curves are shown in Fig. 6.

In the case of Armco iron, S.A.E. 1045 annealed, S.A.E. X4340, and S.A.E. 9260 there is little or no difference shown by the tension test for the un-shot-blasted or shot-blasted conditions. In the case of S.A.E. 1045 for the heat-treated condition where, due to the heat treatment chosen, the residual stresses are assumed to be high, there are some effects shown by shot blasting. This seems to indicate that the superimposing of a residual compressive stress on the outer layers by shot blasting a section already under residual stress due to heat treatment, results in a lowering of the proportional limit, yield point, and tensile strength. As will be seen later this reduction in tensile strength was not in evidence in the case of shot-blasted spring wire.

The fatigue results obtained on each steel are recorded in Table III, summarized in Table IV and plotted in Fig. 7.

In the case of the annealed S.A.E. 1045, and S.A.E. X4340, there are very definite increases in fatigue life

TABLE III.—FATIGUE TEST RESULTS.

Polished Surface		Shot-Blasted Surface	
Stress, psi.	Cycles	Stress, psi.	Cycles
S.A.E. 1045 STEEL (ANNEALED)			
63 600	7 000 broken	64 400	7 000 broken
58 700	10 500 “	58 000	20 200 “
50 900	61 200 “	51 500	59 500 “
47 700	102 700 “	47 700	160 700 “
45 100	191 400 “	45 100	666 300 “
41 900	348 600 “	44 100	1 344 000 “
39 900	2 086 700 “	43 500	43 300 000 not broken
39 200	12 724 000 not broken		
38 600	10 000 000 not broken		
S.A.E. 1045 STEEL (HEAT TREATED)			
102 700	6 200 broken	105 500	9 400 broken
93 500	42 300 “	95 500	39 100 “
84 200	69 200 “	83 700	95 800 “
82 200	417 900 “	79 500	347 400 “
81 400	182 400 “	76 700	543 700 “
81 000	123 000 “	75 000	201 600 “
80 700	10 160 000 not broken	74 000	11 150 000 not broken
77 800	10 148 000 not broken	73 500	10 300 000 not broken
S.A.E. 9260 SILICO-MANGANESE STEEL (HEAT TREATED)			
171 700	13 400 broken		
137 400	65 900 “	189 400	9 500 broken
137 000	47 800 “	167 800	52 100 “
126 200	22 900 “	149 800	305 900 “
116 300	55 700 “	141 100	1 571 400 “
110 900	7 524 500 “	128 600	1 952 400 “
109 400	102 000 “	128 300	3 050 600 “
108 600	11 000 000 not broken	127 500	2 813 200 “
107 600	12 000 000 not broken	126 300	4 725 700 “
		124 700	6 720 100 “
		120 000	6 788 300 “
		109 100	8 996 300 “
		105 900	16 000 000 not broken
ARMCO IRON			
51 300	5 600 broken	40 100	31 700 broken
40 100	37 100 “	32 600	179 200 “
32 800	211 900 “	28 200	2 183 600 “
28 000	1 698 300 “	27 100	4 992 500 “
25 100	11 750 000 not broken	26 800	16 328 000 not broken
S.A.E. X4340 STEEL			
86 000	43 700 broken[a]	102 300	41 100 broken
78 000	119 000 “	85 000	273 700 “
66 000	2 651 000 “		

[a] One inch diameter specimen; the other four specimens ¾ in. in diameter.

TABLE IV.—EFFECT OF SHOT BLASTING ON FATIGUE STRENGTH.

	S.A.E. 1045		S.A.E. 9260	Armco Iron	S.A.E. X4340
	Annealed	Heat Treated			
Diameter of critical section, in.	0.25	0.25	0.20	0.20	¾ and 1
Speed, rpm.	1 750	1 750	1 750	1 750	600
Endurance Limit, psi.:					
Polished surface	39 500	80 700	108 600	26 800	66 000[a]
Shot-blasted surface	43 800	75 000	106 000	27 000	78 000[a]

[a] Probable values.

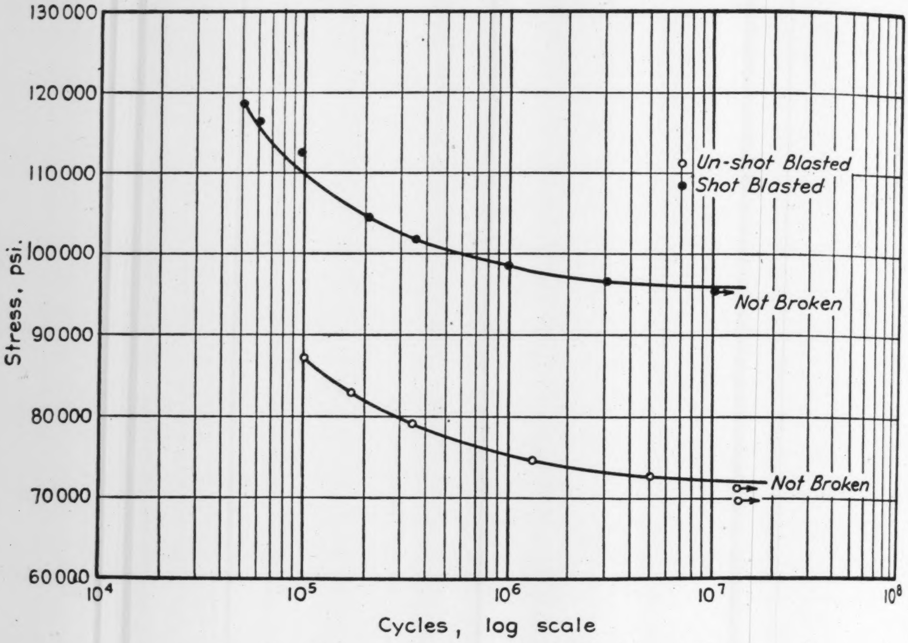


FIG. 8.—Endurance Curves for Unmachined Wire.

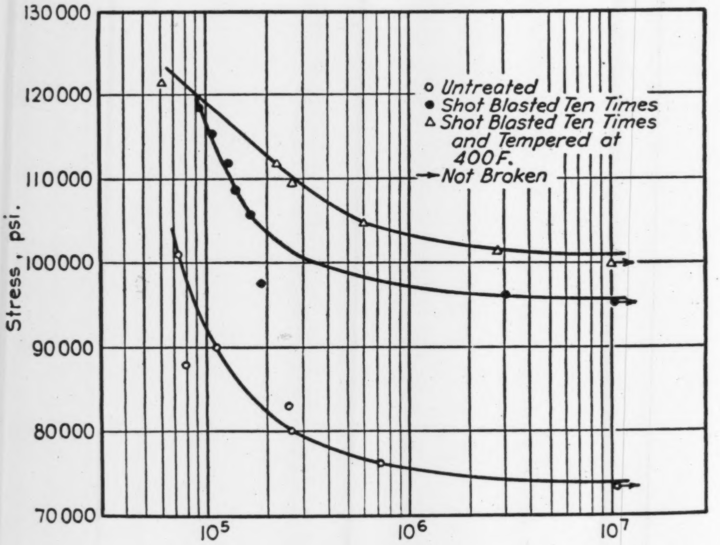


FIG. 9.—Endurance Curves for Unmachined Wire Showing Effect of Tempering After Shot Blasting.

and endurance limits by shot blasting. The increase in endurance limit seems to increase with increase in hardness. The effect of shot blasting the S.A.E. 9260 increases the fatigue life, but makes little or no difference in the endurance limit. For the heat-treated S.A.E. 1045 it should be noted that the hardness is greater than that which would be generally adopted and the endurance limit for the un-shot-blasted condition is higher than the normal value. These high hardnesses chosen for the above two steels were selected in order to obtain a range of hardness irrespective of any residual stresses which might result. The presence of these residual stresses may have prevented any change in endurance limit due to shot blasting.

TABLE V.—FATIGUE TEST RESULTS ON UNMACHINED WIRE SPECIMENS.

Standard Surface Finish		Shot-Blasted Surface Finish	
Stess, psi.	Cycles	Stress, psi.	Cycles
87 200	99 000 broken	118 700	49 000 broken
83 000	170 000 "	116 800	59 000 "
79 400	345 000 "	112 300	95 000 "
74 500	1 313 000 "	104 600	207 000 "
72 600	5 060 000 "	101 500	348 000 "
71 300	12 387 000 not broken	98 200	998 000 "
69 800	12 942 000 not broken	96 600	3 048 000 "
		95 200	10 241 000 not broken

SHOT BLASTING OF UNMACHINED SURFACES

Shot blasting has been applied successfully to certain automobile and aero-engine parts. For a number of years[5] it has been standard practice in this country to shot blast valve springs. Extensive commercial applications for the shot blasting of other springs such as leaf springs have been made by the Eaton Manufacturing Co. Below are given certain results on carbon spring wire. In this work the authors have collaborated with H. H. Clark of that company.

The material tested was domestic wire 0.162 in. in diameter and of the following analysis:

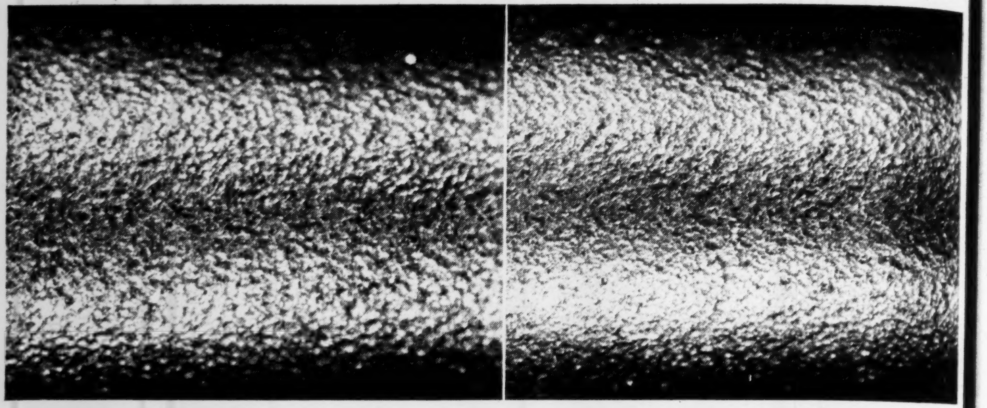
Carbon, per cent	0.65
Manganese, per cent	0.76
Silicon, per cent	0.20
Phosphorus, per cent	0.018
Sulfur, per cent	0.026

Tension and fatigue tests were made on wire samples for the un-shot-blasted and shot-blasted conditions, a Baby Rayflex machine being used for the fatigue tests. The tensile strength was not affected by shot blasting and gave values of 229,500 psi. for both conditions. The fatigue results are recorded in Table V and plotted in Fig. 8. The fatigue strength of the un-shot-blasted wire was ± 72,000 psi. and this was increased to ± 96,000 psi. by shot blasting. This increase in fatigue strength in bending when transformed into equivalent fatigue strength in torsion by use of accepted relationships agreed very well with fatigue tests made by the Eaton Manufacturing Co. on springs from the same wire.

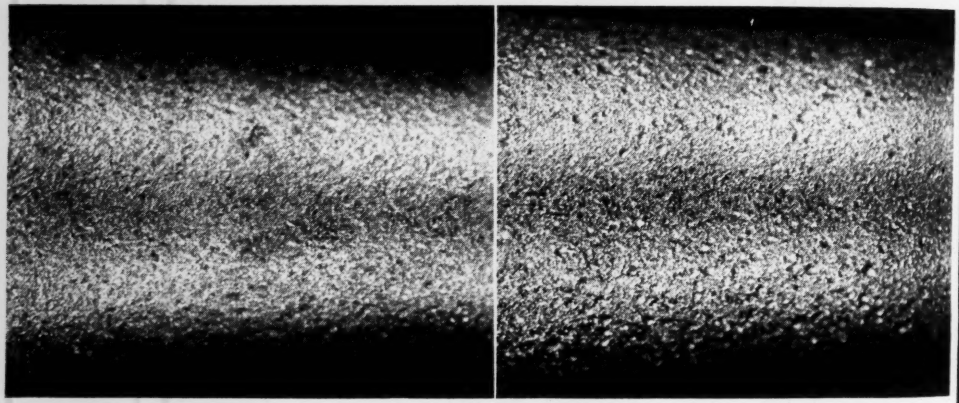
A study was also made on a similar material of the effects of stress relieving after shot blasting. In every case examined, the effect of annealing at 400 F. for 20 min. after shot blasting was to increase the fatigue life as based on the number of cycles. This change can be noted from Fig. 9.

APPEARANCES OF SURFACE

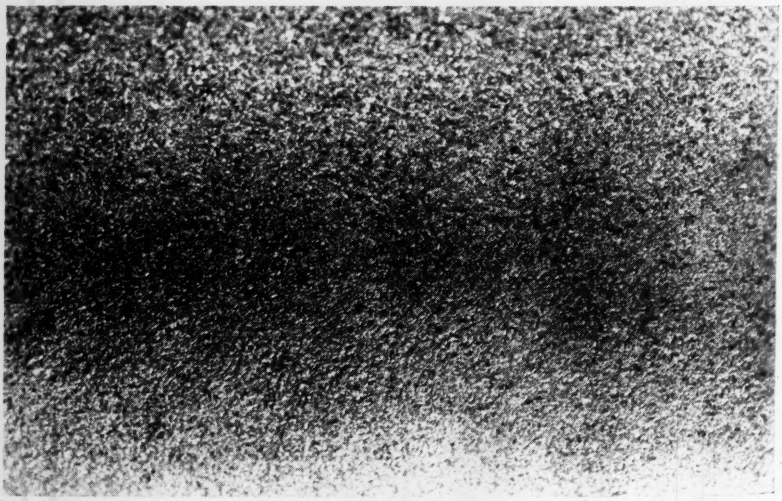
Reference has previously been made to the important part which well-polished surfaces play in developing the full fatigue strength and the decrease which can be expected from unmachined surfaces. The surface after shot blasting however is not smooth but carries very definite markings as a result of the peening action of the steel shot.



SAE 1045 steel (annealed). SAE 1045 steel (heat treated).



SAE 9260 steel Armco iron



SAE ×4340 steel

Fig. 10.—Shot-Blasted Surfaces (×8).

In those applications where shot blasting can be applied, it appears that some modification is necessary of our ideas regarding the necessity of extremely high-polished surfaces so far as the strength is concerned. This does not mean that surfaces with large surface defects can be improved by the shot-blasting process but suggests the negligible influence of small multinotches as obtained in shot blasting.

Figure 10 shows the appearance of the surfaces of the various steels discussed previously in this report. The differences shown may be due to different hardness of the material or to variables in the shot-blasting operation.

TABLE VI.—EFFECT OF QUENCHING STRESSES.

Material	Treatment	Brinell Hardness Number	Tensile Strength, S, psi.	Endurance Limit, S_e psi.	Ratio, $\frac{S_e}{S} = n$	Surface Compressive Stress, psi.
0.42% C.	Normalized. Heated to 1562 F. and quenched in salt solution. Drawn at 210 F.	578	249 000	81 000	0.325	110 000
0.42% C.	Normalized. Heated to 1435 F. and quenched in salt solution. Drawn at 590 F.	477	215 000	98 000	0.45	30 000
1.0% C.	Normalized. Heated to 1472 F. and quenched in salt solution. Drawn at 570 F.	495	240 000	112 000	0.46	negligible

FATIGUE STRENGTH OF QUENCHED-AND-TEMPERED STEELS

General:

The increase in fatigue strength due to shot blasting is held to be due to the compressive stress developed in the outer layers. In order to ascertain whether such a conclusion is tenable a brief review will now be made of residual stresses arising from heat treatment and cold work.

It is generally recognized that these residual stresses are of two kinds, namely, microscopic stress and macroscopic stress. Nadai[6] points out that a classification can be made on the basis of whether the elements in which they occur are comparable in size to the crystal grains, hence microscopic in character, or to the size of the stressed body, hence macroscopic. Very little is known at present as regards the microscopic stresses although we do know that stress relieving of hardened and tempered steel gives a different form of tension test diagram although no structural change has taken place.

Quenching and Tempering:

In quenched-and-tempered steels the macroscopic residual stresses have been measured. Heyn[7] was the first to work on this subject. Using quenched cylinders and measuring the change in length after removing successive surface layers he found that the outside was in compressive stress and the center in tension. Some work has been published by one of the authors[8] on cylinders $1\frac{1}{2}$ in. in diameter by 4 in. long of 0.42 per cent carbon and 1.0 per cent carbon steel. The results are shown in Table VI.

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[6] A. Nadai, "Plasticity," p. 20, McGraw-Hill Book Co., Inc., New York, N. Y.

[7] Martens-Heyn, "Handbuch der Materialenkunde," Second Edition, p. 280, (1912); also E. Heyn, "Einige weitere Mitteilungen über Eigenspannungen und damit zusammenhängende Fragen," *Stahl und Eisen*, p. 442, (1917).

[8] J. M. Lessells, "Fatigue Strength of Hard Steels and Their Relation to Tensile Strength," *Transactions*, Am. Soc. Steel Treating, p. 413 (1927).

Although the surface stress as recorded above was compressive at the outside, when this was decreased by a modification of the heat treatment the endurance limit was increased even though the tensile strength was decreased. Furthermore, while the 0.42 per cent carbon steel with a Brinell hardness of 578 was in a highly stressed condition the 1 per cent carbon steel with a Brinell hardness of 495 had no appreciable residual stress. This seems to suggest some generalization to the effect that a steel may be heat treated to such a high hardness that an abnormal condition is developed so that its fatigue strength may be less than it would be with a reduced hardness.

TABLE VII.—EFFECT OF TEMPERING ON FATIGUE STRENGTH.

Series	Brinell Hardness Number	Tensile Strength, S, psi.	Endurance Limit, S_e, psi.	Ratio, $\frac{S_e}{S} = n$
AA	522	294 200	120 000	0.41
BB	488	282 000	112 000	0.40
CC	433	226 100	98 000	0.43
DD	356	172 900	89 000	0.51
EE	242	148 500	78 000	0.52

TABLE VIII.—EFFECT OF TEMPERING ON FATIGUE STRENGTH.

Tempering Temperature, deg. Fahr.	Tensile Strength, S, psi.	Endurance Limit, S_e, psi.	Ratio, $\frac{S_e}{S} = n$
900	153 000	72 000	0.47
1000	148 000	74 600	0.50
1100	132 000	74 000	0.56

Other data[9] are available in the literature on the effect of tempering on the fatigue strength. Moore published data on a $3\frac{1}{2}$ per cent nickel steel (Table VII). This was annealed, heated to 1450 F., and oil quenched. The series *AA* was not drawn, *BB* was drawn at 400 F., *CC* at 600 F., *DD* at 800 F., and *EE* at 1000 F.

These results indicate that as the tensile strength is decreased by increase in tempering temperature the endurance limit does not decrease as rapidly as evidenced by the increase in ratio n. This increase in the value of n might very well be due to a reduction in residual stress. McAdam[10] published data even more striking on a $3\frac{1}{2}$ per cent nickel steel. The steel was quenched in water from 1450 F. and tempered at 900, 1000, and 1100 F., giving the results shown in Table VIII.

These values again show that as the tempering temperature is increased an increase in the ratio n is to be expected. In this case the actual endurance limit is increased. Extensive data have also been published by Gillett.[11, 12] A nickel-chromium-molybdenum steel of analysis 0.41 per cent carbon, 2.49 per cent nickel, 0.79 per cent chromium, 0.76 per cent molybdenum gave the results shown in Table IX.

TABLE IX.—EFFECT OF TEMPERING ON FATIGUE STRENGTH.

Condition	Brinell Hardness Number	Endurance Limit, psi.
Oil quenched and drawn	400	100 000
Redrawn	385	106 000
Redrawn and slowly cooled	365	112 000

These results show that the endurance limit and endurance ratio can be increased by a suitable tempering operation.

FATIGUE STRENGTH OF COLD-WORKED STEELS

A great amount of experimental data are available on the effect of cold work on the fatigue strength of steels, and since the mechanism of shot blasting is being discussed it seems advisable to review briefly the results which have been made available by various investigators.

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[9] H. F. Moore and T. M. Jasper, "Investigation of Fatigue of Metals," *Bulletin No. 142*, p. 55, University of Illinois.

[10] D. J. McAdam, Jr., "Endurance of Steel Under Repeated Stresses," *Chemical and Metallurgical Engineering*, December 14, 1921; also "Endurance Properties of Steel: Their Relation to Other Physical Properties and to Chemical Composition," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 56 (1923).

[11] H. W. Gillett and E. L. Mack "Molybdenum, Cerium and Related Alloy Steels," Chemical Catalog Co. (1925).

[12] "Prevention of the Failure of Metals Under Repeated Stress," Staff of Battelle Memorial Inst., p. 182, John Wiley and Sons, Inc., New York, N. Y. (1941).

Stretching:

Moore and Kommers[13] investigated the endurance limit of a 0.18 per cent carbon steel after different amounts of cold stretching and obtained a considerable increase, while Horger[14] did similar work on a 0.48 per cent carbon steel and also obtained increases.

TABLE X.—EFFECT OF COLD STRETCHING ON FATIGUE STRENGTH.

Material	Reduction of Area Due to Cold Stretching, per cent	Tensile Strength, S, psi.	Endurance Limit, S_e, psi.	Ratio, $\frac{S_e}{S} = n$
0.18% C	0	61 500	28 000	0.45
	8	67 600	35 000	0.52
	17 to 18	73 400	41 000	0.56
0.48% C	0	88 600	39 800	0.45
	8	111 500	50 000	0.43
	17 to 18	107 000	54 000	0.50

In the case of stretching it can be assumed that the macroscopic stresses are zero. Nevertheless, an increase in endurance limit was experienced for all cases.

Cold Drawing:

For the case of cold drawing references must be made to the work of Brown.[15]

Gill and Goodacre[16] also published important results on different carbon contents for greater degrees of reduction. Where overdrawing was not in evidence increase in endurance limit by reductions from 25 to 90 per cent were found.

In this process, due to the fact that the outer layers are drawn over the underlying ones, the outer fibers are in a state of tensile stress. These investigators also found that there is a definite limit to be reached in cold drawing beyond which the fatigue strength will decrease although the tensile strength is increased. For the 0.86 per cent carbon steel this point was reached at 82 per cent reduction. A further important point was that the endurance ratio was very low varying from 0.32 to 0.22* but that it could be increased by suitable tempering after cold drawing, provided that the reduction by drawing was less than 60 per cent. Beyond this point no benefits in endurance limit or endurance ratio were obtained by tempering.

TABLE XI.—EFFECT OF COLD DRAWING ON FATIGUE STRENGTH.

Material	Reduction of Area Due to Drawing, per cent	Tensile Strength, S, psi.	Endurance Limit, S_e, psi.	Ratio, $\frac{S_e}{S} = n$
0.29% C	0	72 000	34 500	0.48
	11	86 000	39 000	0.45
	23	98 000	40 700	0.41
	35½	104 000	45 000	0.43

Cold Rolling:

Much work has been done on the cold rolling of surfaces. Föppl[17] was the first worker in this field to be followed later by Behrens.[18] Peterson[19] and Horger in this country have since made important contributions to the subject.

Horger[14] reported for a 0.48 per cent

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[13] H. F. Moore and J. B. Kommers, *Bulletin No. 124*, University of Illinois (1921).

[14] O. J. Horger, "Effect of Surface Rolling on the Fatigue Strength of Steel," *Transactions*, Am. Soc. Mechanical Engrs., p. A129 (1935).

[15] R. M. Brown, "An Investigation into Some Effects of Cold Drawing on the Strength of Mild Steel," *Transactions*, Inst. Engrs. and Shipbuilders in Scotland, p. 496 (1927–1928).

[16] F. T. Gill and R. Goodacre, "Some Aspects of the Fatigue Properties of Patented Steel Wires," *Journal*, Iron and Steel Inst., No. 2, p. 143 (1935).

* For heat-treated steels this ratio has been shown to have a value 0.5 to 0.4.

[17] O. Föppl, "Das Drüchen der Oberfläche von Bauteilen aus Stahl," *Stahl und Eisen*, Vol. 49 (1929).

[18] P. Behrens, "Das Oberflächendrüchen dur Erhöhung der Drehschwingungsfestigkeit," Wöhler Institut Braunschweig, Heft 6 (1930).

[19] R. E. Peterson and A. M. Wahl, "Fatigue of Shafts at Fitted Members," *Transactions*, Am. Soc. Mechanical Engrs., p. A1 (1935).

carbon steel increases of 24 to 32 per cent in fatigue strength. The depth of the cold-worked material varied from 0.006 to 0.015 in. depending on the normal roller pressure.

In this case the stresses on the outside are compressive.

Shot Blasting:

In the case of shot blasting a high compressive stress is developed in the outer skin. The depth affected by the shot blast is very small, being as previously indicated approximately 0.006 in. on a 1-in. diameter specimen.

TABLE XII.—SUMMARY OF THE EFFECT OF VARIOUS PROCESSES.

Condition	Type of Surface Stress	Depth of Cold Working, in.	Effect on Endurance Limit
Quenched and tempered	Compressive	......	Decrease
Stretched	None	Entire surface	Increase
Cold drawn	Tensile	......	Increase
Cold rolled	Compressive	0.006 to 0.015	Increase
Shot blasted	Compressive	0.006	Increase

RESIDUAL STRESS *Versus* COLD WORKING

In the light of these results it is possible to summarize the effects of these various processes. This is done in Table XII.

As to a probable cause for the effect of such processes as cold drawing or cold rolling on the endurance limit there are several schools of thought. Föppl[20] believes that the increase is due to deformation of the surface and consequent hardening, while Thum[21] believes it is due to compressive stresses in the outer layers. It is somewhat difficult to accept the viewpoint of Thum since, as previously discussed, in a quenched-and-tempered steel high residual compressive stresses may exist if the hardness is carried beyond a certain limit. Under such stress condition the endurance limit value may be considerably lowered. In no case examined were such stresses found to be favorable. On the other hand in the case of drawn rod or wire the residual stresses at the outside are tensile. Nevertheless, provided no overdrawing exists, increase in fatigue strength can be expected. Obviously these occurrences which are well supported by experimental evidence would be reversed if stress were the chief governing factor, since we would expect a compressive stress on the outside to be beneficial in fatigue, and conversely a tensile stress on the outside to be detrimental.

Consideration of the these facts leads to the conclusion that the shot-blasting process produces a cold working of the outer surface which is the main contributing factor to increase in fatigue strength. Nevertheless, the results may also be influenced by the stress distribution and the increase in fatigue strength may be due to a combination of cold work and residual stress.

DISCUSSION

In the light of the experimental data given in this paper and the results of previous work by Zimmerli, it is seen that under certain conditions considerable increase in fatigue life and endurance limits can be expected from shot blasting. This is true for all the steels tested but in the case of those steels which have high residual stress due to quenching and insufficient tempering this increase on endurance limit may not be in evidence. Evidently when the high compressive stresses on the outside due to quenching have compressive stresses due to shot blasting superimposed there is created a condition in which the cold working of the surface has little or no effect. At least the available experimental evidence seems to point that way.

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[20] O. Föppl and A. Thum, *Zeitschrift* des Vereines Deutscher Ingenieure, pp. 77, 1335, 1337 (1933).

O. Föppl, "Oberflächendrüchen," *Mitteilungen* des Wöhler Institut (1935).

[21] A. Thum and W. Bautz, "Ursachen des Steigerung der Dauerhaltbarkeit gedrückter Stabe," *Forschung des Ingenieurwesens*, p. 121 (1935).

Previous research has indicated that a smooth and highly polished surface is necessary for high fatigue strength. This now requires some modification because in the case of shot blasting the surfaces are slightly roughened.

CONCLUSIONS

The conclusions which can be drawn from this work are as follows:

1. Considerable increase in fatigue life and endurance limits of steels can under certain conditions be obtained by shot blasting the surface. By inference similar advantages may be gained for metals other than steel.
2. The increase in fatigue life and endurance limits seems to be due to a cold working of the outer surface although the resulting compressive stress may be beneficial.
3. There does not appear to be any advantage as regards endurance limits in shot blasting surfaces where high residual stresses due to quenching and insufficient drawing are present.
4. The beneficial effects can be removed by annealing but annealing at low temperatures, provided this is not sufficiently high, is beneficial as regards fatigue life for shot-blasted surfaces.

DISCUSSION

MR. F. P. ZIMMERLI[1] (*presented in written form*).—The calling of the shot blasting process to the attention of the Society cannot be too highly commended, since almost everyone can use it to advantage.

We agree with the authors that Herbert was really the first one to call attention to the possibilities of the process. It is unfortunate that his commercial applications were of but slight interest at the time. Had it been otherwise, the design of many parts would by this time be much changed and improved. In our own instance, we are grateful to Mr. Herbert for the start his paper gave our laboratories in the research we have since accomplished.

The results that Mr. Almen obtained showing 0.006 in. depth of blasting effect seem a reasonable check with our own microscopic findings which we reported as positive evidence down to at least 0.004 in. Below this depth we could see no structural difference, which does not mean it was absent.

The methods of shot blasting are as the authors have stated. The use of 25-lb. air pressure, however, does seem low. All the machines with which we are familiar need at least 60 lb. and even then are slow to produce the desired effect, 100 psi. being much better.

The authors' remarks on the effects of cold drawing confirm our commercial tests. Wire that has had over 80 per cent reduction since the last patenting or heat treatment is usually overdrawn and worthless, even in music wire grades. It is interesting to note in this regard that for springs made of present-day music wire or oil-tempered valve spring wire, when the coiling stresses are properly relieved, both have equal endurance limits despite different tensile strengths. If the same wire is hardened and tempered after coiling, instead of being a pretempered wire, the pretempered wire will show a higher endurance limit. This is because steel is treated at the mill in a continuous process. This process heats the wire to the quenching temperature, then the wire runs through oil and directly in the draw, which is usually molten lead. The material runs through so fast that the steel enters the draw heat at a temperature well up to the flash point of the oil. The result is a material with a greater reduction of area for a given tensile strength, or, conversely, a higher tensile strength for a given reduction of area. The endurance limit is better and so are the usual twist tests. We believe response to shot blasting has been augmented and that research work could develop a pretempered wire whose response to shot blasting might be even better. In passing, it is interesting to note that pretempered wire is forbidden in many airplane engine valve springs. This often results in receiving springs not of the highest possible quality although good enough for the performance necessary.

We are in agreement with the authors' first conclusion, but with the second one we would like to ask why, if the increase in fatigue is only due to cold work, we shot-blast springs used in bending on the tension side only for maximum fatigue life. In a quenched piece of steel there is no way of knowing the compressive stress distribution and in music wire the compressive stresses due to the dies are accompanied by other stresses. Can we not conceive of shot blasting putting in compressive stress much more uniformly than any other process listed with consequently more uniform fatigue life? It seems to us that both work hardening and the prestressed condition are responsible as we pointed out in our paper.[2]

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[1] Chief Engineer, Barnes-Gibson-Raymond, Division of Associated Spring Corp., Detroit, Mich.

Conclusion three is undoubtedly accurate from a laboratory point of view but needs, we feel, some elaboration. As a commercial example, our factory is producing certain firearm springs around 55 Rockwell "C" scale. The fatigue life of these parts has been more than quadrupled by shot blasting with fine ($\frac{1}{64}$-in.) shot. The heat treatment of the parts is an oil quench, followed by a 425 to 450 F. draw. In this instance we feel the shot blasting has removed sharp edges and other stress raisers, and rounded out fillets a bit in addition to any value of its own. These are points all producers should consider in regard to the shot blasting process.

MR. H. H. CLARK[3] (*presented in written form*).—The shot blasting of springs and of kindred articles has been responsible for phenomenal increases in fatigue life and endurance limit. The success which has resulted from shot blasting has led us to conclude that the process is always good. We find nothing in the comprehensive and objective treatment of the subject by the authors which is at variance with our conclusions; but certain of the remarks make it apparent that the maximum benefits of shot blasting are obtained only when sound stock and satisfactory heat treatment are used.

The data presented in the paper to show the comparison between fatigue life and endurance limit for polished and shot-blasted surfaces, respectively, transcend the balance of the data in timely importance. The infinitely greater productivity of the shot-blasting operation as compared to that of polishing should be given proper consideration wherever high-strength design problems are encountered.

The authors indicate that in the presence of high residual quenching stresses shot blasting is of dubious advantage. A study of the data presented in Table VI compared with that in Table IV for heat-treated S.A.E. 1045 and S.A.E. 9260 leads one to the conclusion that where very hard steels are concerned, an increase in the drawing temperature is more beneficial than shot blasting where endurance limit is concerned. This conclusion, however, leads one to conjecture. If the increased drawing temperature is sufficient to relieve the residual stresses, would not subsequent shot blasting enable one to obtain a worth-while increase in endurance limit over that obtained by the increased drawing temperature? Of course, this is a matter of degree, but in the final analysis, it is important to investigate such a question.

In considering the curves shown for S.A.E. 9260 steel in Fig. 6, we are prompted to remark in the interest of emphasis that, though the difference in endurance limit between the shot-blasted and polished specimens is of small order, the shot-blasted specimens have many times the life of the polished specimens at stress values above the endurance limit. To a lesser extent the same is true for heat-treated S.A.E. 1045 steel.

Where the difference in endurance limit is small and other considerations are equal, the material and treatment giving the greater life at stresses above the endurance limit in most cases would be preferable. Beyond this consideration, one should recognize that a casual scratch may prove much more harmful to a polished surface than to a shot-blasted surface where the multinotch effect of the shot impressions will mitigate the harmful effects of the scratch.

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[2] F. P. Zimmerli, "Shot Blasting and Its Effects on Fatigue Life," *Preprint No. 51*, Am. Soc. Metals (1940).

[3] Manager, Coil Spring Department, Spring Division, Eaton Manufacturing Co., Detroit, Mich.

In the successful application of shot blasting, it is desirable to provide means whereby as much of the shot as possible impinges the surface of the material under treatment at an inclination angle of 90 deg. In addition, it is well to consider the degree of impingement desired at various locations. Depending upon the design of the piece to be shot blasted, it is conceivable that a differential rather than a uniform impingement is preferable.

As an example, in the case of valve springs we shot blast the springs individually in the successive stations of a conveyor arrangement which rolls the springs through the shot stream, the spring axis being at right angles to the major axis of the shot stream. Analysis of the cycloidal motion thus imparted to the spring reveals that there is a differential action of the shot stream on the surface of the wire. The impingement on the inside diameter, where the torsional stress is maximum, is substantially greater than on the outside diameter, a most desirable condition.

Another advantage in this method for shot blasting valve springs is that each ray of shot in the shot stream at some point in the travel of each spring impinges the surface of the wire at 90 deg. This condition promotes the efficient use of the blast stream. The method also has the obvious advantage of assuring the maximum uniformity of treatment from spring to spring in a particular lot.

It is highly important to control the shot-blasting process. Test strips described by the authors, photographs, and surface analyzers are helpful in checking the final results. In arriving at satisfactory results, the amount of shot used per unit time, the condition of the shot, and the amount of work must all be controlled. Of these items we find that the condition of the shot is the most difficult to control, and we supplement the mechanical arrangements for this control by periodic screen analyses of the shot.

MR. OSCAR J. HORGER.[4]—The authors show how one may increase fatigue strength on unmachined members, but on machined members we are very much surprised at the small relative increase in fatigue strength or endurance limit, especially with the S.A.E. 1045 steel.

There appears to be little practical merit in shot blasting machined plain specimens in view of the reported small increase in fatigue strength. In general, much greater increase in fatigue strength would be obtained through cold working by the process of running a burnishing roller under pressure over the member. This may mean that by a further study of shot size, shot pressure, and impingement of shot and other factors, proper control of shot-blasting conditions may be secured for obtaining optimum fatigue strength.

It was mentioned that shot blasting was used to increase the fatigue strength of crankshafts. It would be valuable to know what increase was obtained since crankshafts involve the stress concentration from fillets. It has been our experience that cold working of members with stress concentration gives larger increases in fatigue strength than is obtained on plain members without stress concentration. Can the authors substantiate this statement with reference to shot blasting?

One might expect the results which

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[4] In Charge of Railway Engineering and Research, Timken Roller Bearing Co., Canton, Ohio.

the authors obtained on silico-manganese steel. The high hardness and ultimate strength of this material and the fact that the yield point is close to the ultimate strength would indicate a small capacity for cold working. For this reason little increase in fatigue strength may be expected due to shot blasting. On the other hand, if increased fatigue strength from shot blasting is a result of favorable surface residual compressive stresses, then some increase in fatigue strength may be expected independent of capacity for cold working. It would be worth while knowing the state of residual stress in the silico-manganese bars before and after shot blasting. Such knowledge may give information on the controversial question of whether the increased fatigue strength usually found by cold working is due to residual stresses or structural changes. The authors may have investigated this condition.

Visual inspection of the line widths of the X-ray diffraction patterns in Fig. 3 would seem to indicate plotted points different from those shown in Fig. 4. The width of line for 0.009 in. below the surface appears wider than for the line 0.006 in. below the surface. Also, in view of the line diffusion near the surface, it would be interesting to know by what method the authors measured these lines, even if a microphotometer was used.

MR. R. E. PETERSON.[5]—It will be of interest to compare the manner of failure of shot-blasted specimens with that of nitrided specimens. Fatigue tests of nitrided specimens[6] have shown that failure occurs beneath the nitrided layer. If we indicate the depth of the layer by δ and the endurance limit of the unnitrided material by σ_e (accompanying Fig. 1), then the percentage increase in fatigue strength which one might expect in an unnotched bending specimen due to nitriding would be:

$$\frac{\Delta\sigma_e(100)}{\sigma_e} = \frac{100}{\dfrac{d}{2\delta} - 1}.$$

The above-mentioned test data[6] showed percentage increases in rather good agreement with the above calculation, although somewhat greater in some cases. It is suggested that similar calculations be made for the shot-blasted specimens. In the nitrided specimens the source of failure could be clearly seen as a small "fish-eye" similar to that noticed in weld tests. Did the shot-blasted specimens show anything similar?

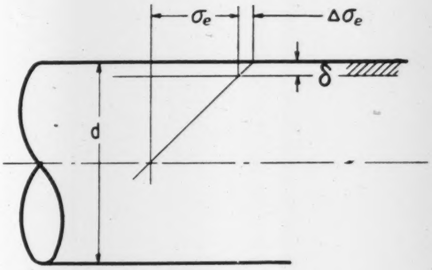

FIG. 1.

MR. T. J. DOLAN[7] (*by letter*).—Shot blasting is one of several methods of surface treating metal members that is effective in increasing their fatigue strength. Other processes that also involve only localized surface treatments but have been shown to improve the fatigue strength of steel might include nitriding, case carburizing, cyaniding, cold rolling, and flame or induction heating methods of surface hardening. However, shot blasting seems to offer a simple method of treatment that will produce beneficial results without requiring the use of special steels, elaborate heating or processing equipment, or close mechanical and metallurgical control of the treatment. The authors are to be commended for their further contributions to the solution of this intriguing problem of increasing the fatigue strength of highly stressed metal members.

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[5] Manager, Mechanics Division, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[6] O. Hengstenberg and R. Mailaender, "Biegeschwingungsfestigkeit von nitrierten Staehlen," *Zeitschrift*, Vereines Deutscher Ingenieure, Vol. 74, p. 1126 (1930).

[7] Assistant Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

The writer would like to comment mainly on the effects of the roughened surface finish on the fatigue test results presented for the shot-blasted specimens, since the surface pitting undoubtedly had considerable influence on the strengths exhibited by the specimens. Perhaps four interacting factors control the fatigue strength of the shot-blasted specimens, namely,

1. The state of residual stress induced in the surface fibers of the specimens;
2. The change in mechanical strength properties of the surface metal caused by the cold working;
3. The "stress raising" or notching effect of the indentations left on the surface by the shot-blasting process; and
4. The "notch sensitivity," or relative resistance the metal offers to the damaging effects produced by the localized stresses developed at these surface indentations.

The authors have pointed out that item 2 above is perhaps more important than item 1 in increasing the fatigue strength. However, if we assume a case for which the residual compressive stress existing in the surface fibers is equal to the magnitude of the alternating stress superimposed by the fatigue testing machine, the resultant cycle of stress resisted by the surface fibers would vary from zero to a maximum stress in compression. When subjected to this type of cyclical stress variation (or any cycle in which the mean stress in the cycle is a compressive stress), a metal will resist a greater alternating range of stress without failure than for those cases in which the mean stress in the cycle is a tensile stress. Hence, large residual compressive stresses should aid somewhat in increasing the fatigue strength of the member.

The roughened surface shown in Fig. 10 of the paper would normally lower the fatigue strength of these specimens to values considerably below the endurance limits obtained for *polished* specimens if the surface fibers were not greatly strengthened by the shot-blasting operation. For instance, McAdam[8] has shown that for steel specimens roughened on the surface by small hemispherical corrosion pits the fatigue strength of the specimen is cut approximately in half. The writer would like to emphasize, therefore, that even though several of the shot-blasted steels did not show endurance limits greater than those of the polished specimens, they did exhibit much greater strength than that of unpolished specimens for the case of the spring wire, and would also be expected to be greatly superior to specimens with ordinary machined or roughened surfaces having about the same depths of indentations as those on the shot-blasted specimens.

Since actual members in service are rarely polished as perfectly as the standard polished fatigue specimens, we never expect actual machine parts to exhibit as great a fatigue strength as would normally be obtained from highly polished laboratory test specimens. However, the authors' data indicate that if the surfaces of machined parts were shot blasted, we could then expect to obtain fatigue strengths of the finished members that were as high as those obtained from polished laboratory specimens. Thus it appears that shot blasting should prove useful in applications where it is impractical to polish the finished parts carefully, and would offer a means of obtaining a greatly increased fatigue strength over that obtained from the relatively rough machined surfaces.

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[8] D. J. McAdam, Jr., and G. W. Geil, "Pitting and Its Effect on the Fatigue Limit of Steels Corroded Under Various Conditions," see p. 696.

It is a recognized fact that fine-grained quenched-and-tempered alloy steels having relatively high endurance limits normally exhibit greater notch sensitivity than steels which have not been subjected to hardening operations. For some steels test results show a great difference between the endurance limits of notched and unnotched specimens, whereas for other steels or for other heat treatments only a small difference is obtained. This variation in notch sensitivity is shown in the accompanying Table I which lists flexural fatigue endurance limits obtained by the writer for two steels subjected to several heat treatments. These data illustrate the fact that even though various treatments may increase the fatigue strength of *polished* specimens, the increased notch sensitivity in the stronger steels may be enough to prevent any marked increase in fatigue strength of specimens containing stress raisers. For instance, the fatigue strength of unnotched (polished) specimens of the hot-rolled S.A.E. 3140 steel was increased about 40 per cent by quenching and tempering, whereas the fatigue strength of specimens containing a small transverse drilled hole remained unchanged by this heat treatment. Similarly, the fatigue strength of the quenched-and-tempered S.A.E. 1045 steel shown in the table was much greater than the fatigue strength of the same steel in the normalized condition. Yet the fatigue strength of the quenched-and-tempered specimens containing a notch was actually smaller than that of the normalized specimens containing a notch. Evidently the notch sensitivity of the steels tested by the authors either was not increased markedly by shot blasting or the increase in strength of the surface metal was sufficient to bring the over-all strength of the shot-blasted specimens with roughened surfaces up to that of the polished specimens.

TABLE I.

Material	Endurance Limit, psi.		Stress concentration Factor, K[b]
	Unnotched	Notched[a]	
S.A.E. 1045 + lead, cold-drawn	48 000	19 000	2.53
S.A.E. 1045 + lead, normalized	41 500	26 000	1.60
S.A.E. 1045 + lead, quenched-and-tempered	63 000	20 000	3.15
S.A.E. 3140, hot-rolled	64 000	31 000	2.06
S.A.E. 3140, quenched-and-tempered	90 000	31 000	2.90

[a] Specimen 0.40 in. in diameter, with a transverse drilled hole 0.04 in. in diameter.

[b] $K = \frac{\text{Endurance limit unnotched specimen}}{\text{Endurance limit notched specimen}}$.

In connection with the tension test diagram shown in Fig. 6 of the paper, the writer would like to inquire whether the authors found a great variability in the tensile properties of different specimens of the annealed S.A.E. 1045 steel. In this diagram it appears that the two polished specimens exhibited quite different shapes of stress-strain curve and one apparently exhibited a definite yield point, whereas the other did not. Also in connection with the fatigue tests there was a considerable difference in the hardness of the various steels tested, and it would be interesting to know what relative depth of cold working was produced by the shot blasting in the various types of steel tested, since this may have considerable influence on the results obtained. The appearance of the pits shown in Fig. 10 indicates somewhat different depths of indentations caused by the steel shot, and these indentations in some of the softer steels may be of the same order of magnitude as the depths of cold working reported by the authors for the S.A.E. X4340 steel. One would normally expect that with materials of different hardness and ductility, all shot blasted in the same manner, the relative depths of cold working would be quite different, and hence their fatigue strengths probably would be affected by different relative amounts.

MR. T. McLEAN JASPER[9] *(by letter)*.—This new investigation by Lessells and Murray suggests that shot blasting may be used as a substitute for polishing. If this is true, then it will result in a great reduction of expensive polishing operations for the finishing of all but bearing surfaces of machinery. The reason for this is that it puts the surface material in considerable compression. Since the high tensile stresses of service will come below the surface, and since failure never occurs in compression but in tension or shear, this setting of the surface in initial compression has the effect of producing the most highly stressed zone in tension or shear, with no surface at all for breakdown.

There is one phenomenon that the writer observed which would tend to reduce the value of initial stresses: When many cycles of stress have been applied, it was found that internal stresses were very much reduced. This leads one to ask: "Would service fatigue, in time, reduce the beneficial effect of compression and, in this manner, nullify some of the effects of shot blasting?"

MESSRS. J. M. LESSELLS[10] AND W. M. MURRAY[10] *(authors' closure)*.—In regard to Mr. Zimmerli's comments on the second conclusion, the authors are in agreement, but unfortunately there seems to be no simple way of determining the relative influence between the effects produced by building up a thin layer of cold-worked material to better resist the stressing and the change produced in the stress distribution due to the superimposition of compression. It does not seem that this would change the uniformity of the fatigue life except from the point of view of perhaps peening small surface scratches, which undoubtedly would tend to produce a scattering of the test results.

Since most of the discussors make some comment about either the cold working of the surface or the presence of a surface compressive stress influencing the results, it will be well to point out that any benefits which may be derived from either of these effects will be dependent upon the stress gradient as one moves from the surface. In other words, one must consider a race between two effects as one proceeds into the material (and this applies to regions of stress concentration as well as to uniform sections), the dissipation of the stress and the dissipation of any beneficial effects produced by the surface treatment. If the stress dissipates more rapidly than the strengthening, due to the shot blasting, then one can expect the fatigue results to be higher than for the corresponding polished surface. These benefits may be modified by the mode of stressing. The data in the paper only discuss fatigue in bending. In the case of direct fatigue stress, the outer compressive layer will decrease the maximum tension on these layers, and while for equilibrium there must be a tensile stress on the inside, this, as shown in Fig. 5, may be too small to affect the results. Consequently, shot blasting may be beneficial if the fatigue stresses are produced by direct stress instead of bending.

Mr. Peterson's remarks are of some considerable significance. The relation he gives can now be applied to certain additional experimental data obtained by the authors since the paper was written.

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[9] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

[10] Associate Professor of Mechanical Engineering, and Assistant Professor of Mechanical Engineering, respectively, Massachusetts Institute of Technology, Cambridge, Mass.

σ_e = Endurance limit for S.A.E. X4340 steel, 1 in. in diameter, un-shot blasted and without stress concentration = ±64,000 psi.

σ_e = Endurance limit for S.A.E. X4340 steel, 1 in. in diameter, shot blasted and without stress concentration = ±70,000 psi.

$$\frac{\Delta\sigma_e(100)}{\sigma_e} = 9.4 \text{ per cent}$$

According to Hengstenberg and Mailaender and assuming that the depth affected is 0.006 in. = δ.

$$\frac{\Delta\sigma_e(100)}{\sigma_e} = \frac{100}{\dfrac{d}{2\delta} - 1} = \frac{100}{\dfrac{1}{2 \times 0.006} - 1} = 1.2$$

Fracture of the tapered specimen at a cross-section removed from the critical one will change the above value very little. These results may indicate that the residual stress is quite an important factor in the improvement obtained by shot blasting. For the case of nitriding, the relationship seems to agree better with experimental results. Here δ = 0.020 in. and $\frac{\Delta\sigma_e(100)}{\sigma_e}$ = 5 per cent on the basis of known data. By theory

$$\frac{\Delta\sigma_e(100)}{\sigma_e} = \frac{100}{\dfrac{0.75}{2 \times 0.025} - 1} = 7\,\% \text{ (approx.)}$$

It should be noted, however, that the effect of such surface treatment will be most beneficial where the stress gradient is high, and consequently from the point of view of increasing fatigue resistance, will be best applied to sections with stress concentration or to small cross-sections. This is a subject which is obtaining some further attention by the authors.

The authors regret that Mr. Horger seems to be disappointed with the results obtained from shot blasting, especially S.A.E. 1045. It should be emphasized that the purpose of the paper was to study the effect of shot blasting for an ascending order of Brinell hardness, and it so happened that to obtain the hardness desired: namely, 311, the S.A.E. 1045 steel was in a high state of residual stress and had a hardness beyond the commercial range for such material. This does not mean that this material, heat treated to a lower hardness of 250, would not also show considerable benefits from shot blasting. Furthermore, the advantages of shot blasting, as pointed out in the paper, are not merely increased fatigue strength, but equal or increased fatigue strength at reduced cost, since polishing is eliminated in the case of machined parts.

In regard to Horger's comments with regard to the line widths obtained from the X-ray analysis, it should be pointed out that these indicate a trend and that the apparent variation between the lines corresponding to depths of 0.006 in. and 0.009 in. is of little significance, since conditions at both these locations are approximately the same as in the remainder of the base metal. The widths of the lines were measured by means of a microphotometer.

In replying to Mr. Peterson's question about the appearance of the fractures, it may be said that what he refers to as the "fish-eye" was also observed for shot-blasted specimens with both large and small diameters, without stress concentration.

In replying to Mr. Jasper's question, the authors regret that they have no information covering tests of a duration much beyond 10,000,000 cycles. It seems that the answer can only be found by making some tests of very long duration.

FATIGUE COMPARISON OF 7-IN. DIAMETER SOLID AND TUBULAR AXLES

By O. J. Horger[1] and T. V. Buckwalter[1]

Synopsis

Rotating cantilever beam fatigue tests were made on axles about 7 in. in diameter using steel of about S.A.E. 1045 analysis. Comparison of axle fatigue resistance due to a press-fitted wheel was made between "as-forged" solid members and seamless tubes in "hot-rolled" and several conditions of heat treatment.

Results showed that tubular axles having high tensile strength values may or may not exhibit greater fatigue strength than those having lower physical properties. Observations on the effect of residual stresses are given. Some tubular axles showed greater fatigue strength than the solid ones.

The object of this experimental investigation was to determine the fatigue resistance of solid *versus* tubular type axles for railroad car service. Solid axles tested represented conditions of manufacture, design, and material consistent with existing railroad specifications and these tests[2] were made for the Association of American Railroads under the direction of an Axle Committee headed by Chairman W. I. Cantley, Mechanical Engineer, Association of American Railroads. The tubular axles investigated were of two types; one of which was tested with permission of A.A.R. and the results reported by the manufacturer[3]; the other was tested as a separate project in order to contribute information on certain questions pertaining to the fatigue strength of axles.

Some apparent engineering advantages of tubular over solid axles are:

(*a*) Lower weight—about one-third less.

(*b*) Less unsprung weight which reduces impact stresses.

(*c*) Thin sections may offer better opportunity for heat treatment and the retention of favorable residual stresses.

The work reported here is concerned with the fatigue strength of the axle in the portion where the wheel is pressed on. The large stress concentration introduced in the axle at the position where the wheel is mounted results in greatly reduced axle fatigue resistance and as a consequence much research is in progress in an attempt to improve this weakness.

Axles tested represent specimens which could be applied to a car. The test of full-size members gives direct data bearing on the questions of processing conditions, size effect, residual stresses, and variables associated with large members which become factors of unknown influence when scale-size specimens are tested.

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[1] In Charge of Railway Engineering and Research, and Vice-President, respectively, The Timken Roller Bearing Co., Canton, Ohio.

[2] "Passenger Car Axle Tests 5½ × 10-in. Journals," Fourth Progress Report, Assn. Am. Railroads, Chicago, Ill., April 1, 1940.

[3] Discussed in bulletin entitled "Tubular Railway Axle" issued by Pittsburgh Steel Co., Pittsburgh, Pa. (1940).

TABLE I.—CHEMICAL COMPOSITION AND PROPERTIES OF SAMPLES OF THE TEST AXLES.

	Chemical Composition, per cent						
	Carbon	Manganese	Phosphorus	Nickel	Chromium	Sulfur	Silicon
Solid Axle[a]	0.48 to 0.52	0.60	0.023	...	...	0.024	0.20
Tubular Axle *A*	0.37	0.80	0.018	0.09	0.04	0.020	0.26
Tubular Axle *B*	0.50	0.73	0.017	0.25	0.13	0.015	0.27

PHYSICAL PROPERTIES.

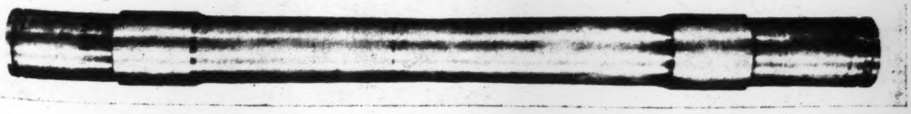
	Solid Axle	Tubular Axle A			Tubular Axle B	
	½ in. Diameter by 2 in. Gage Length Midway Between Center and Outside Diameter	Sub-size Specimens ¼ in. Diameter by 1 in. Gage Length			½ in. Diameter by 2 in. Gage Length	
					Center of Wall Thickness	
		Outside Diameter	Center	Inside Diameter	Hot Rolled	Water Quenched and Tempered
Yield strength, psi.[b]	44 500–65 000	82 700–93 900	73 600–87 700	80 600–86 800	61 000–63 500	72 000–81 000
Tensile strength, psi.	90 500–107 000	113 200–115 100	109 100–113 100	102 000–103 900	103 500–106 000	119 000–120 000
Reduction of area, per cent	24.8 to 39.1	40.8 to 42.8	42.9 to 45.0	46.9 to 53.1	41.9 to 42.2	47.2 to 50.6
Elongation, per cent.	16.0 to 24.0	19.0 to 20.0	20.0 to 21.0	22.0 to 24.0	22.0 to 23.0	20.5 to 21.5
Brinell hardness (end tensile specimen)	179 to 212	229 to 335	223 to 235	217	183 to 192	217 to 223
Izod impact, ft-lb.	7.0 to 15.0	....	....	....	13 to 16	17 to 23

[a] A.A.R. Specification M-101-36, grade B, Manual of Standard and Recommended Practice, Assn. Am. Railroads Mechanical Division, Chicago, Ill. (1936).

[b] Determined by drop-of-beam method.

Solid type axle conventionally used.

Tubular axle—type A.



Tubular axle—type B.

FIG. 1.—Three Types of Axle Tests.

MATERIAL

Axles from three materials were tested where the analysis conformed essentially to S.A.E. 1040 and 1045 steels. Table I shows the chemical analysis and physical properties of samples of the test axles. References covering the material in the wheels mounted on the test axles are

TABLE II.—RESULTS OF FATIGUE TEST.

Axle	Wheel Mounting Conditions				Calculated Axle Bending Stress at Inside Hub Face, psi.		Axle Life		Remarks as to Axle Condition in Wheel Fit After Test[a]
	Wheel Hub		Wheel Fit on Axle, in.	Tonnage	Solid Axle	Hollow Axle	Revolutions, millions	Equivalent miles on 36-in. Wheels	
	Diameter, in.	Stand, in.							
SOLID AXLES—AS-FORGED CONDITION									
9-C-2	11	½	0.0077	85	19 000	....	8.13	14 460	Broken
11-B-2	11	½	0.0082	95	19 000	....	1.75	3 130	Cracked 0.200 in. deep (79 per cent)
11-A-2	11	½	0.0077	120	17 000	....	10.90	19 450	Broken
8-A-1	11	½	0.0072	95	15 000	....	70.65	125 700	Broken
8-B-1	11	½	0.0077	125	15 000	....	40.29	71 700	Broken
10-C-2	11	½	0.0075	95	13 500	....	84.25	150 000	Cracked 0.015 in. deep (6 per cent)
10-A-1	11	½	0.0075	95	13 500	....	84.3	150 000	Cracked 0.027 in. deep (2 per cent)
3-B-1	11	½	0.0085	95	13 500	....	171.5	305 000	Cracked 0.023 in. deep (25 per cent)
4-B-1	11	½	0.0087	100	13 500	....	170.5	304 000	Not cracked
8-A-2	11	½	0.0075	105	12 000	....	84.3	150 000	Not cracked
24-B-1	11	½	0.0085	95	12 000	....	152.5	272 000	Not cracked
18-F-1	11	½	0.0082	85	12 000	....	171.7	306 000	Not cracked
HOLLOW AXLE A—WATER QUENCHED ON OUTSIDE DIAMETER AND TEMPERED									
7	10	....	0.0073	70	30 000	32 100	0.29	514	Not cracked[b]
11	10	....	0.0070	85	19 000	20 100	87.2	155 500	Cracked 0.033 in. deep (55 per cent)
18	10	....	0.0075	75	17 000	18 200	88.0	156 700	Cracked 0.013 in. deep
14	10	....	0.0065	75	17 000	18 100	88.8	158 400	Cracked (Fig. 7)
3	10	....	0.0070	65	15 000	16 100	87.1	155 500	Cracked
14A	10	....	0.0077	75	15 000	16 000	87.7	156 300	Cracked 0.024 in. deep
24	10	....	0.0055	70	13 500	14 450	88.5	158 000	Not cracked
25	10	....	0.0069	90	13 500	14 350	88.5	158 000	Not cracked
2	10	....	0.0060	80	12 000	12 800	84.7	151 000	Not cracked
8	10	....	0.0067	80	12 000	12 850	84.7	151 000	Not cracked
HOLLOW AXLE B—HOT-ROLLED CONDITION									
372	10	½	0.0080	80	19 000	21 700	1.07	1 910	Broken
373	10	½	0.0113	90	17 000	19 400	0.68	1 210	Broken (Fig. 6)
371	10	⅛	0.0115	110	13 500	15 300	6.90	12 300	Broken
374	10	⅛	0.0103	95	12 000	13 500	18.01	32 100	In test
HOLLOW AXLE B—WATER QUENCHED ON OUTSIDE DIAMETER AND INSIDE DIAMETER AND TEMPERED									
376(*a*)	10	½	0.0115	100	19 000	21 500	0.40	708	Broken
375	10	⅛	0.0120	115	13 500	15 200	4.87	8 690	Broken
378	10	⅛	0.0107	103	12 000	13 700	4.20	7 480	Broken
HOLLOW AXLE B—FLAME HARDENED WHEEL SEAT ONLY									
381	11	⅛	0.0163	120	19 000	21 600	89.1	158 700	Not cracked
382	11	⅛	0.0123	150	19 000	21 500	99.6	175 500	Not cracked
381A	11	⅛	0.0140	90	22 000	25 180	85.7	152 600	Not cracked

[a] Figure in brackets following "cracked" indicates percentage of the circumferential length of wheel seat which is fatigue cracked by one or more disconnected short cracks. Depth of crack was measured where it was believed to be a maximum.

[b] Broke in body portion of axle, outside of wheel fit where surface is not machined, at calculated bending stress 31,100 psi.

found in A.A.R. Specifications M-107-36[4] or A.S.T.M. Specifications for Multiple-Wear Wrought Steel Wheels (A 57 – 39).[5] Scleroscope hardness of these wheels was 35 to 37 as measured on the end of the hub face midway between the bore and outside diameter of the hub.

AXLE AND WHEEL ASSEMBLIES TESTED

Design of members tested is shown in Figs. 1, 3, 4, and 5. One design of solid axle conventionally used on some new cars being built and two designs of tubular axles, types *A* and *B*, are reported in this investigation. A wheel is press fitted on one end of the axle in accordance with usual railroad mounting practice and the rim of this wheel also serves as a means of attaching the axle assembly to the testing machine. Wheel fits and mounting tonnages on axles are detailed in Table II. A definite mixture[4] of white lead and linseed oil was painted on the axle and wheel bore previous to mounting.

AXLE PROCESSING

Processing details as to rolling, forging, and machining of solid axles were previously published.[6]

Tubular axle *A* was formed from a $7\frac{1}{4}$ in. outside diameter tube, made on a Pilger mill, having a nominal wall thickness of 1 in. Each end was upset to obtain a nominal $1\frac{3}{4}$-in. wall thickness in the region of the wheel fit and particularly at the location of the inside end of the wheel hub face. Diameter of the axle was turned to 7 in. at the wheel fit. Heat treatment consisted of water quenching only on the outside of the tube. This was followed by drawing at 600 F.

Tubular axle *B* was machined from a $7\frac{3}{4}$-in. outside diameter tube, made on a Mannesman mill, having a nominal wall thickness of $1\frac{5}{8}$ in. with no intermediate forging or forming operations. Some of these axles were tested in the (*a*) hot-rolled condition; (*b*) normalized at 1650 F., reheated to 1500 F. and quenched in water for 6 min. with the ends of the tube partially capped which limited the flow of water through the bore, and then tempered at 1050 F. and allowed to cool in still air; and (*c*) normalized at 1650 F. and the wheel seat only flame hardened.

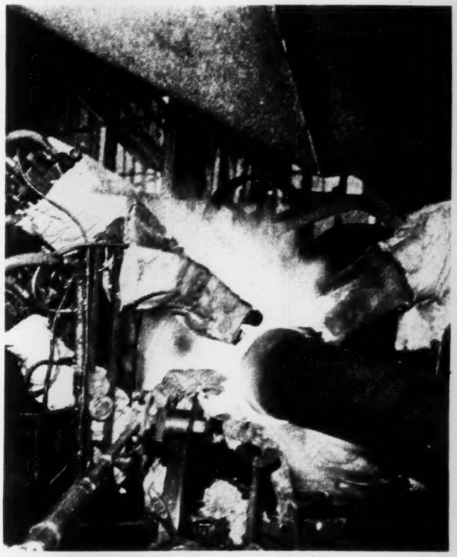


FIG. 2.—Method of Flame Hardening Axle.

Flame hardening of the wheel seat surface was accomplished[7] by rotating the axle at 110 rpm. in a field of four equally spaced flame hardening heads as shown in Fig. 2. Heating time varied from 2 min. to 2 min., 30 sec. after which the surface was water-spray quenched and then drawn in oil at 350 F. for 1 hr. Scleroscope surface hardness was 30 before flame hardening and varied from 50 to 70 after flame treatment. This surface was ground for the wheel fit removing about $\frac{1}{32}$ in. on the diameter but the hardness of this ground surface was not measured.

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[4] Manual of Standard and Recommended Practice, Assn. Am. Railroads, Mechanical Division, Chicago, Ill. (1936).

[5] 1939 Book of A.S.T.M. Standards, Part I, p. 201.

[6] O. J. Horger and H. R. Neifert, "Fatigue Strength of Machined Forgings 6 to 7 in. in Diameter," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 723 (1939).

[7] Flame hardening done by Linde Air Products Co. under direction of H. V. Inskeep.

TEST CONDITIONS

These fatigue tests were made on large double-end rotating cantilever beam machines previously described.[6] The speed of test was 1450 to 1500 rpm. for both solid axles and type *A* tubular axles while type *B* tubular axles were tested on a larger machine operating at 600 to 800 rpm. The nominally calculated maximum bending stress as it is distributed over the length of the test axles is shown adjoining the axle sketch in Figs. 3, 4, and 5.

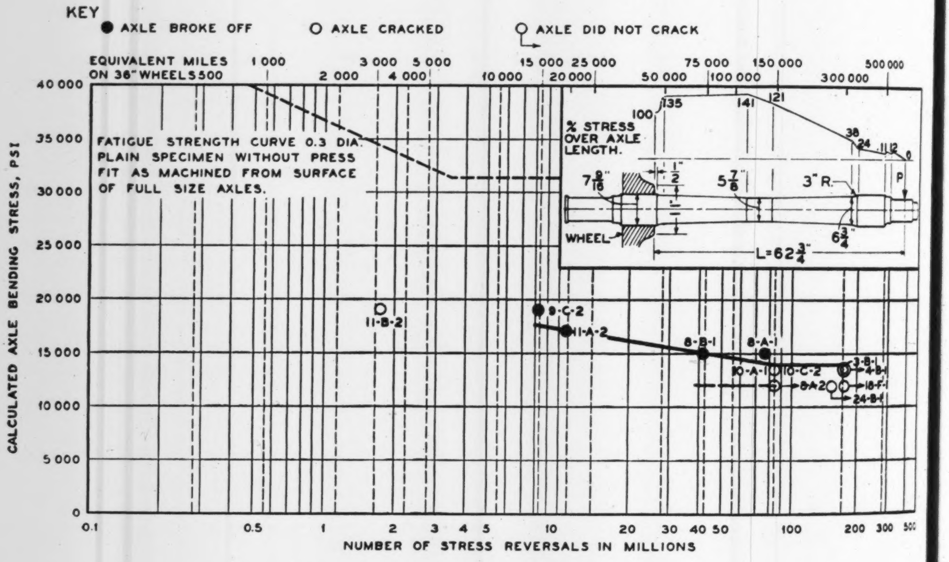


FIG. 3.—*S-N* Data on Solid Type Axles.

Axle bending stress values, *S*, given throughout this paper refer to the stress as calculated by the elementary beam formula:

$$S = \frac{P\,L}{Z}$$

where the symbols are all shown in Figs. 3, 4, and 5 except for *Z* which is the axle section modulus at the wheel seat. The bending stress for the case of the tubular axles is given in Table II for the section modulus of the axle considered as both a solid section and the actual hollow section. This procedure is done for convenience in comparing the solid and hollow axles discussed later.

The endurance limit values are based on 84.3 million revolutions which is equivalent to 150,000 miles on 36-in. diameter wheels. The arbitrary practice is followed of establishing the endurance limit as a stress 1000 psi. below the lowest stress at which any axle broke off and 1500 psi. below the lowest stress at which fatigue cracks were found to initiate.

A total of 32 axles was tested of which 12 were of the solid type and 20 were hollow axles. Results of these fatigue tests are shown in Table II. These data are plotted in the usual *S*–*N* curves of Figs. 3, 4, and 5 on the basis of the stress being shown as though the hollow axles had solid sections.

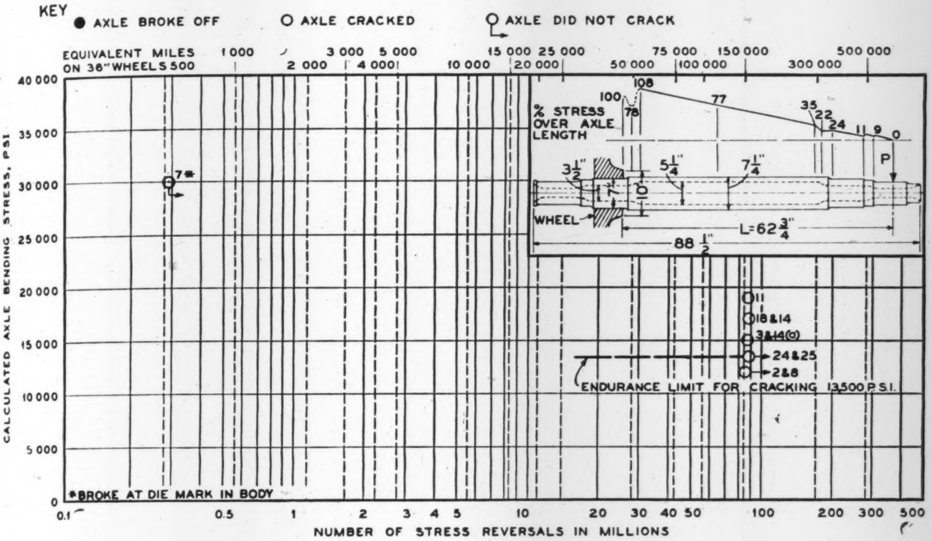


FIG. 4.—*S-N* Data on Tubular Axles Type A.

Stress given on basis of solid section – see Table II and text for stress in tubular section.

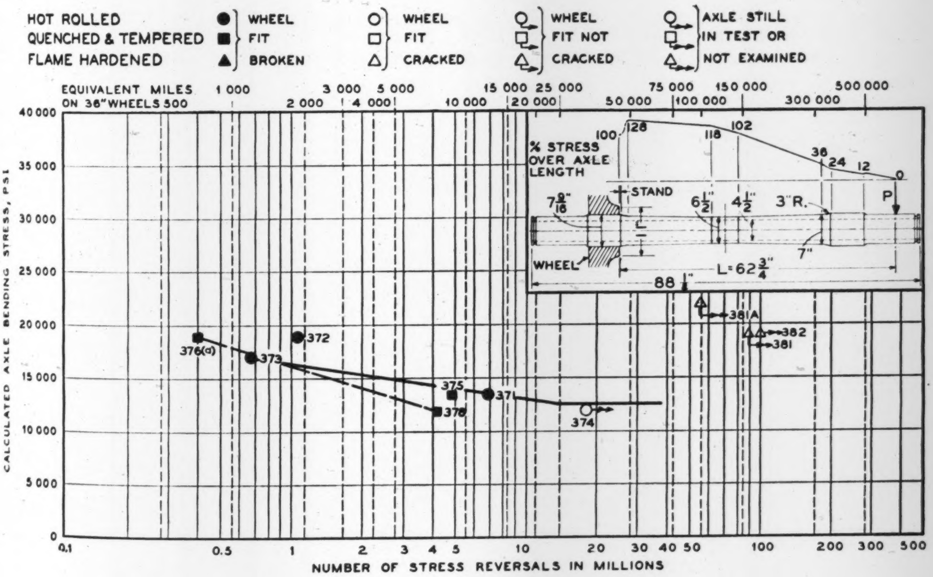


FIG. 5.—*S-N* Data on Tubular Axles Type B.

Stress given on basis of solid section—see Table II and text for stress in tubular section.

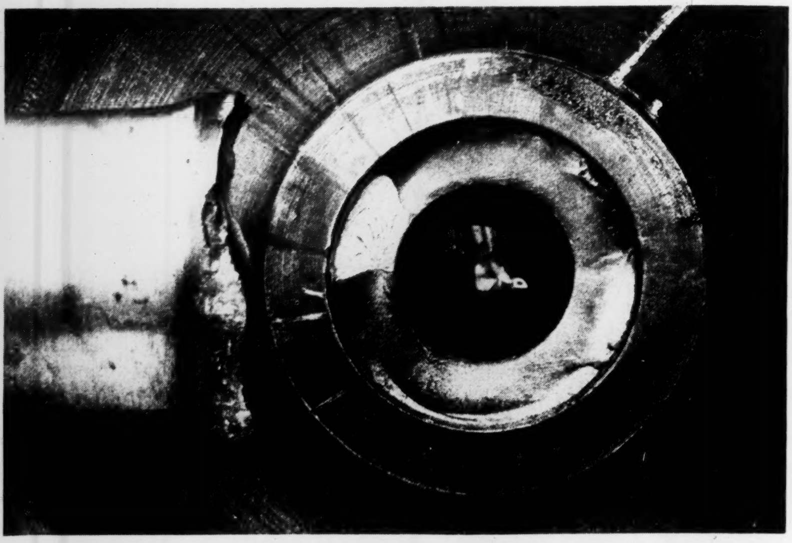


FIG. 6.—Tubular Axle Broken Off in Wheel Fit (Axle No. 373).

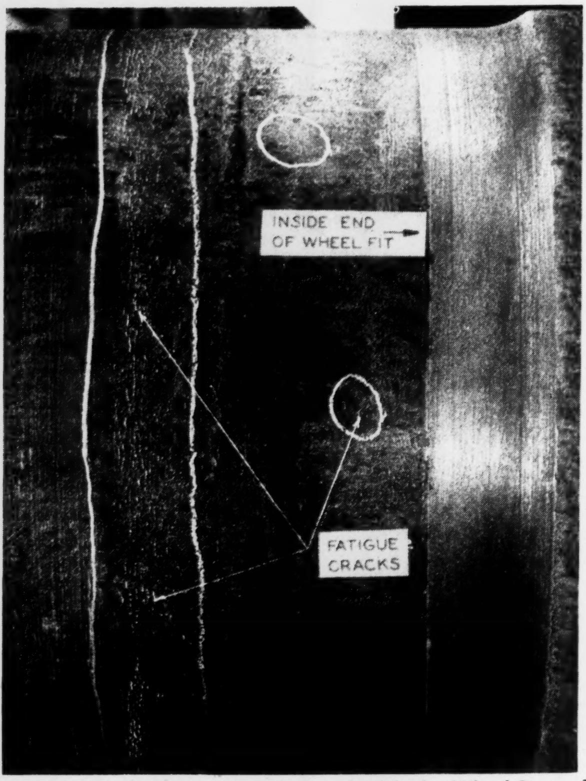
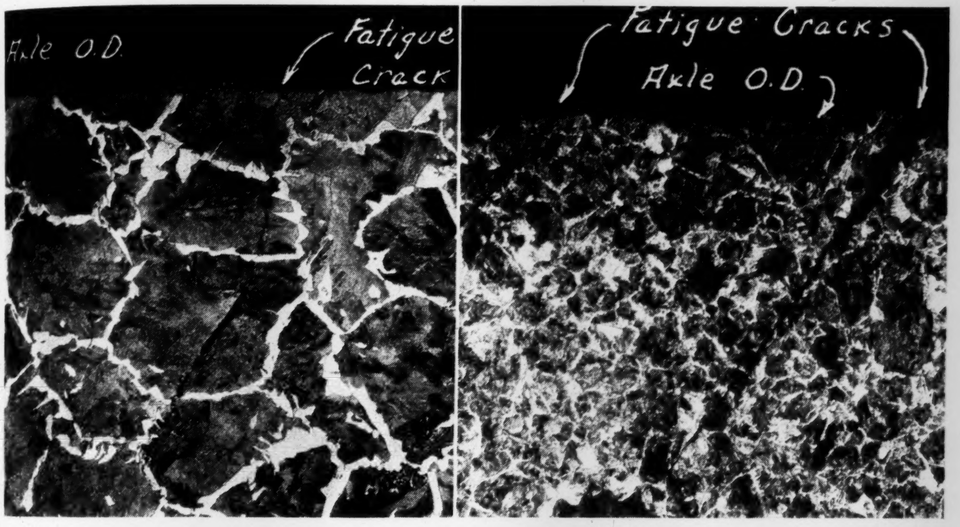
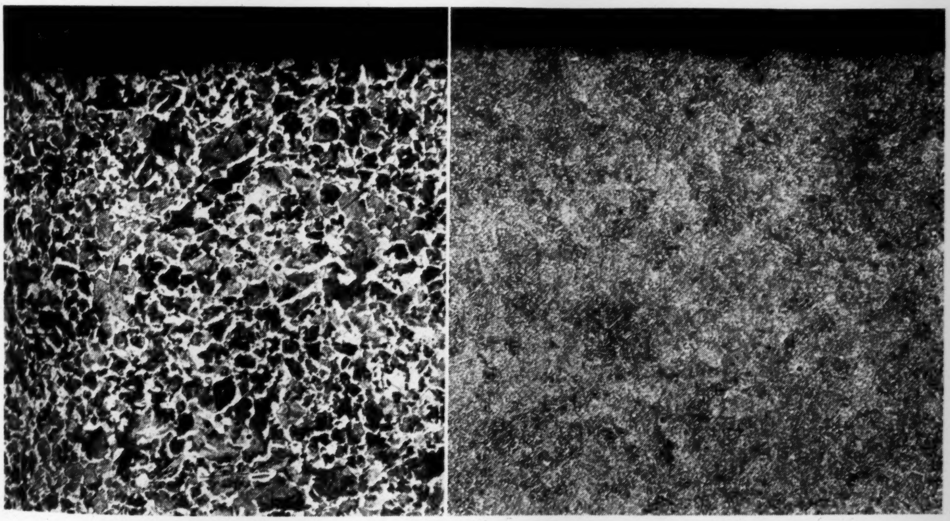

INSIDE END OF WHEEL FIT
FATIGUE CRACKS

FIG. 7.—Wheel Seat of Tubular Axle After Test and with the Wheel Pressed Off for Magnaflux Examination. Note numerous short failure fatigue cracks included within marked regions of wheel fit (Axle No. 14).

Typical failed hollow axles are shown in Figs. 6 and 7 and photomicrographs of most materials tested are shown in Fig. 8.



(a) Solid axles as forged (No. 3 B-1). (b) Tubular axle type A, quenched and tempered (No. 144).



(c) Tubular axle type B, hot rolled (No. 371). (d) Tubular axle type B, quenched and tempered (No. 376).

FIG. 8.—Photomicrographs of Longitudinal Section of Various Test Axles Taken at Wheel Seat Surface (×100).

DISCUSSION OF TEST RESULTS

A summary statement of all fatigue tests is presented in Fig. 11. In studying this figure it is important to recognize that the stress limits are given on the basis of considering the tubular axles as having solid sections. This consideration is reasonable and necessary for comparison purposes if we assume the axle forces produced in service to be the same whether a hollow or solid axle is used.

Therefore Fig. 11, besides indicating endurance limit values, also expresses the comparison of the load-carrying capacities of the wheel seats of the various axles when the solid and hollow axles have the same outside diameter.

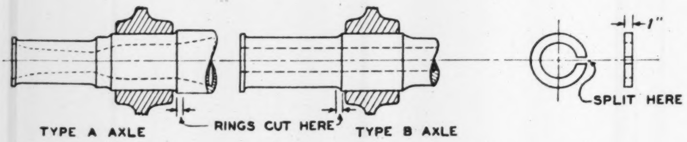


FIG. 9.—Location of Rings Cut Out of Tubular Axles.

No evaluation can be made, from the type of fatigue tests reported here, as to the influence that the lower unsprung weight and changes in axle rigidity of the tubular over the solid axle would have on the impact stresses in the axle in service.

The exact reason for the large differences in fatigue values exhibited for the various axles in Fig. 11 is not always apparent, but the following discussion is presented.

Residual Stresses:

Bühler and Buchholtz[8] have made an investigation of interest in connection with these axle tests in that they correlated fatigue values of plain specimens with the magnitude of residual stresses present in their test members. Briefly summarizing some of their results they found that (*a*) initial compressive stresses on the surface gave an increase in fatigue strength over the case of no residual stresses, (*b*) initial tensile stresses on the surface gave a lower fatigue strength than the case of no residual stresses, (*c*) favorable residual stresses could be obtained by heat treatment with little change in the tensile strength of the material, and (*d*) the magnitude of initial residual stresses is decreased due to repeated stressing. It has been known for some time that residual compressive stresses on the surface of members, with or without stress concentration, obtained by cold working leads to improved fatigue values over parts not cold worked.

An attempt was therefore made to obtain an indication of the presence of residual tangential stresses in several of the tubular axles tested. Transverse slices about 1 in. thick were cut out of several tubular axles and split as shown in Fig. 9. A decrease in diameter of the rings is considered an indication of tangential compressive stresses at the surface of the tubes. Results in Table III indicate that type *A* tubular axles had

TABLE III.—INDICATION OF RESIDUAL TANGENTIAL STRESSES IN SEVERAL TUBULAR AXLES.

	Tubular Axle Type A		Tubular Axle Type B	
	Water Quenched on Outside Diameter and Tempered		Hot Rolled	Water Quenched on Outside Diameter and Inside Diameter and Tempered
	Axle No. 14A	Axle No. 18	Axle No. 372	Axle No. 375
Decrease in average diameter of ring after splitting, in.	0.0328	0.0343	0.0034	0.0053
Axle stress, psi. Section Considered as:				
Solid	15 000	17 000	19 000	13 500
Tubular	16 000	18 200	21 700	15 200
Revolutions, millions	87.7	87.9	1.07	4.87
Remarks about axle wheel fit after fatigue test	Cracked 0.024 in. deep	Cracked 0.013 in. deep	Broken	Broken

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[8] H. Bühler and H. Buchholtz, "Effect of Residual Stresses on the Bending Fatigue Strength," *Stahl und Eisen,* Vol. 53, p. 1330 (1933)

much higher residual compressive stresses than type *B*, and this condition may be responsible for their better performance. Also on other tubular axles tested, but not reported here, the diameter of the rings increased a measurable amount indicating residual tensile stresses on the surface. These latter axles exhibited considerably lower fatigue strength values than the type *A* axles. may be expected to be larger than is apparent from Table III; in the case of type *B* axles less change between the actual and measured values may result. While only a qualitative measure of the residual tangential stresses is given in Table III, it is equally if not more important to also measure the longitudinal stresses. Such heat treatments as were used here, however, which produce residual tangential compressive stresses at the surface are often accompanied by longitudinal compressive stresses. Further investigation is under way to obtain accurate determination of residual stresses in all three planes and the above observations are given only as information.

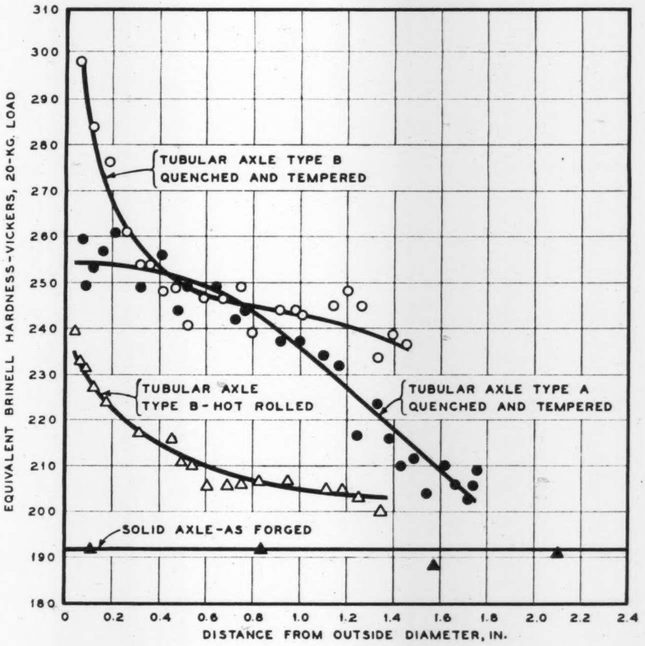

FIG. 10.—Hardness Curves Through Axle Sections.

The above qualitative study of residual stresses was made on the tubes after their fatigue test because of a shortage of specimens but such analysis should be made on members before the fatigue test for the reason mentioned in (*d*) above. It was necessary to take the slices from the type *A* tubes from a region of the axle subjected to much higher stresses than was the case for the *B* axles as indicated in Fig. 9. For this reason the residual stresses for the type *A* axles, if measured before fatigue test,

Hardness survey through several tubular sections is typically represented in Fig. 10, and the hardness gradient may have some significance as to the presence of residual stresses.

HOT-ROLLED *versus* QUENCHED-AND-TEMPERED TUBES

While complete S–N curves were not obtained for type B tubular axles it is interesting to note from Table II and Fig. 5 that the hot-rolled tubes gave at least as long a life in the region tested above the endurance limit as similar quenched-and-tempered axles. In fact Fig. 5 indicates the possibility of a lower endurance limit value for the latter treatment except that the number of tests made were not sufficient to establish an endurance limit for 84 million stress reversals. From a comparison of the physical properties (Table I) and hardness curves (Fig. 10) of the quenched-and-tempered tubes with the hot-rolled the former treatment may be expected to render greater fatigue resistance than the latter, which was not in accordance with the findings. Comparison of micrographs in Fig. 8 (*c*) and (*d*) are of interest in this connection.

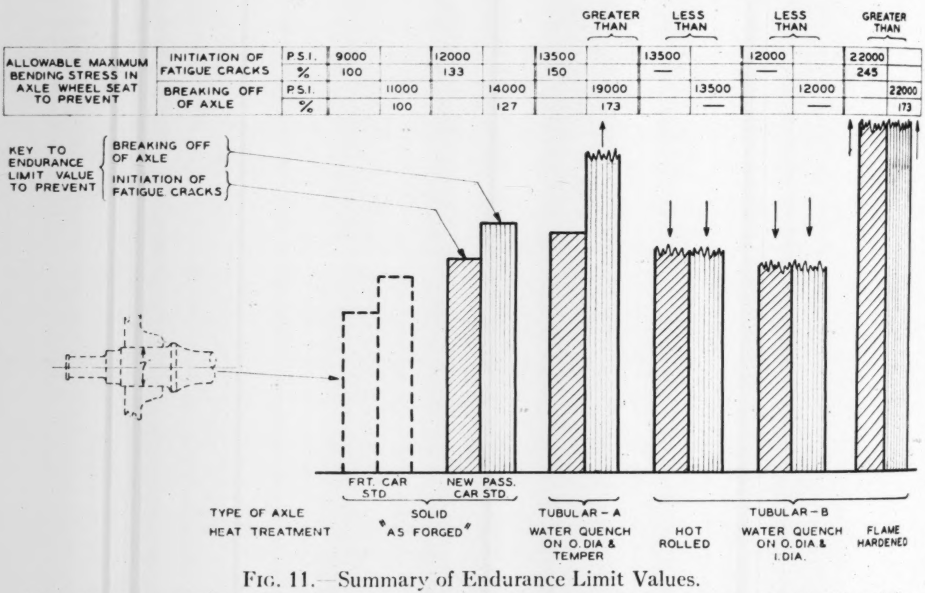

FIG. 11.—Summary of Endurance Limit Values.

Stress given here on basis of solid sections so as to express comparison in load-carrying capacities of solid and hollow axles having same outside diameters. Stress for actual hollow section tested is about 7 per cent greater than is shown for type A tubular axle and 14 per cent for type B

Dotted portion of diagram shown for comparison purposes and data taken from previously published data[2] for axle shape shown and 0.47 to 0.54 per cent plain carbon steel in "as forged" condition.

FLAME-HARDENED AXLES

Fatigue tests on scale-size axles 2 in. in diameter were previously[9] published showing that large improvements in axle fatigue resistance could be obtained by flame hardening the wheel fit portion of the axle. While the tests reported here on the three full-size axles tested are not many in number (Table II and Fig. 5) the available results tend to confirm those found in the earlier investigation of 2-in. flame-hardened members. Figure 11 shows that of all the axle tests reported here the highest fatigue resistance to the initiation of fatigue cracks in the axle wheel fit was obtained with the flame-hardened axle wheel seat.

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[9] O. J. Horger and T. V. Buckwalter, "Fatigue Strength of 2-in. Diameter Axles with Surfaces Metal Coated and Flame Hardened," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 733 (1940).

CRACK DETECTION

The method used to determine whether fatigue cracks are present in the axle wheel fit following the repeated stressing is important because it determines the endurance limit value at which fatigue cracks will not initiate. Examination of all axles reported here was made by the dry magnaflux method with magnaflux equipment type KR3 operated at a maximum of 3000 amp. current and using No. 1 white powder.

The wet method of magnaflux examination is more sensitive than the dry system and instances are known where the latter procedure revealed the presence of cracks $\frac{1}{4}$ in. long by $\frac{1}{64}$ in. deep which were not found by the former method. Even the use of the wet method leaves some question as to whether the indications of cracks obtained by this procedure are actually fatigue cracks. This question arises because the region of the axle wheel fit in which fatigue cracks develop presents a roughened surface along with a sluffing and scoring of metal due to the minute working of the wheel on the axle. Observations with a microscope are of some assistance but not positive. A simple expedient in the case of these test members would be to etch the surface and the presence of cracks will be revealed. This etching, however, is not always looked upon with favor. For this reason pieces may be cut out of the axle where crack indications are found by the wet method and micrographic examination made to determine whether cracks are actually present. Such a procedure was not followed in these tests in order to establish the endurance limit value.

CONCLUSIONS

Tests reported here represent a progress report on the study of the fatigue strength of the wheel-fit portion of railroad car axles. Figure 11 shows that tubular axles may be made which will have greater fatigue resistance than solid axles of the type now being used in railroad service. Sensitivity of crack detection method used to examine the axle after test is important in establishing fatigue values given in this paper.

Acknowledgment:

Appreciation is expressed here to H. R. Neifert of the Timken Research Laboratory for carrying out these fatigue tests.

DISCUSSION

Mr. L. B. Jones[1] (*presented in written orm*).—This paper makes another valuable contribution to our information on the accelerated fatigue behavior of axles. The effect of initial compressive stresses in the surface of the wheel fit, whether produced by the forging operation, by cold rolling, or by flame hardening, seems to be quite well established so far as this form of fatigue testing is concerned. Just how such surface treatment will affect the impact strength is not so fully established.

In ordinary forgings the relation between the tensile and impact properties of the material is pretty definite, so much so that the impact test has fallen into disuse, and many metallurgists feel that the drop testing of axles could be discontinued with impunity. This test was introduced for the purpose of guarding against high residual forging stresses. It is generally recognized that cold working, or any other form of residual stress in the material, has an adverse effect on impact strength, particularly at reduced temperature. On a railroad, the necessity for the use of material which will withstand moderately low temperature without serious loss of strength is always present.

In addition to their study of the tubular axle, the authors make reference in this paper to previous experiments with a metallized sleeve at the wheel fit. It is to be hoped that they will be able to supplement their further experiments with tubular axles by tests with metallized sleeves on full-sized axle specimens. This method of improving fatigue life, if successful, has the advantage that it does not involve localized changes in the material structure of the axles.

Mr. R. E. Peterson.[2]—The authors call attention to an interesting comparison of quenched-and-tempered and hot-rolled materials for a case (tubular axles B) where residual stresses have been shown to be practically absent. The former gave better physical properties as shown by tension and hardness tests but the latter gave better fatigue strength when tested as a press-fitted axle. The authors also state that the micrographs of Fig. 8 (*c*) and (*d*) are of interest in this connection. The hot-rolled material is quite coarse grained while the quenched-and-tempered material is very fine grained. The latter condition is generally associated with higher sensitivity to stress concentration. It is suggested that to test this effect by itself, completely apart from the axle fit problem, two series of 0.3-in. diameter beam specimens be made up from the above two materials: (1) conventional specimens, (2) specimens with a circumferential groove of about $\frac{1}{32}$-in. contour radius. With conventional specimens, the quenched-and-tempered material will show a decidedly higher endurance limit, but with the grooved specimens the endurance limits will be closer together, and the hot-rolled material may actually be slightly higher.

The residual stress studies made

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[1] Engineer of Tests, Test Dept., The Pennsylvania Railroad Co., Altoona, Pa.

[2] Manager, Mechanics Division, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

appear to account for the difference of results of tubular axles of type *A* and *B*. The remarkably high endurance strengths obtained under special conditions should be of considerable engineering importance.

MR. O. J. HORGER.[3]—The discussion by Jones raises the question as to the possible adverse effect on impact strength resulting in axles from surface treatments and residual stresses described in this paper. As stated by Jones, the customary drop test made on full-size axles has fallen into disuse; in this connection it should be noted that impact tests made on Izod specimens machined from full-size solid axles give a wide range of impact strength values (5 to 25 ft-lb.). There is no evidence that axles having low impact values give less satisfactory service operation than axles having high impact values. There is definite evidence from axle fatigue tests indicating no correlation between Izod impact values and fatigue strength determined for full-size axles.

I would appear that there is little or no engineering evidence to establish the need for impact strength in axles. This statement could possibly be extended to apply to rails. The heavy cold working of the surface of the rail head in service produces residual compressive stresses which are apparently beneficial rather than detrimental to rail life. It would therefore appear from inference that the proper kind of residual stresses in axles would not be dangerous from the standpoint of impact.

A different type of small specimen than that suggested by Peterson is now under fatigue test; 1/25 scale size axles are being machined for fatigue test from the full-size axles. In this way the influence of both stress concentration and rubbing corrosion in the wheel fit will be determined for these small specimens. It is hoped that when these tests are completed some additional data will be available as to the effects of size, structure, and residual stresses upon fatigue strength.

Some precautions must be taken when small specimens are machined from full-size members having noticeable hardness gradient as shown for the treated axles in Fig. 10. In such tests it would be necessary (*1*) to mark the side of the specimen containing the outer fibers of the full-size axle, (*2*) to note whether fatigue failure of the small specimen initiated from the outer or inner fibers of the full-size axle from which it was machined, and (*3*) to evaluate fatigue data on the basis of the tensile properties of the fibers in which failure initiated.

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[3] In Charge of Railway Engineering and Research, The Timken Roller Bearing Co., Canton, Ohio.

PITTING AND ITS EFFECT ON THE FATIGUE LIMIT OF STEELS CORRODED UNDER VARIOUS CONDITIONS

By D. J. McAdam, Jr.,[1] and G. W. Geil[1]

Synopsis

A study is made of the form, size and distribution of the pits caused by corrosion of steels in a stream of well water, a stream of Severn River water, a stream of distilled water, a spray of softened water, and a spray of normal salt solution. The results of this study are correlated with diagrams representing the lowering of the fatigue limit due to the corrosion. Pits in these steels tend to be roughly hemispherical at first, but they change to saucerlike form, and spread irregularly along the surface. This change is more rapid with carbon steels than with alloy steels, especially 3½ per cent nickel steel. Carbon steels, especially during corrosion in salt spray, tend to form sharp rootlike projections at the bottoms of rounded corrosion pits. The tendency to form such projections, however, depends somewhat on the chemical composition; it is less for alloy steels than for carbon steel.

Consideration is given to the theoretical stress concentration factor as influenced by the form and distribution of the corrosion pits, and to the effective stress concentration factor as influenced by the form and size of the pits and by the notch sensitivity of the metal.

Cyclic stress accelerates corrosion pitting. When the combined influence of stress, cycle frequency, and corrosion time is sufficiently great, the cyclic stress causes transverse extension of round or irregular pits, with development of projections of the form of crevices or fissures. When the combined influence of these three variables is somewhat less, the cyclic stress has no appreciable effect on the form, but tends to increase the size, especially of a few of the pits. These effects of cyclic stress on the forms and sizes of the pits account for the observed lowering of the fatigue limit.

The influence of simultaneous stress and corrosion on the fatigue limit and the influence of cyclic stress on corrosion have been discussed by the senior author in previous papers. In the first five of these papers (**2** to **6**)[2] the cyclic stress conditions were such as to cause failure by fatigue (corrosion-fatigue). In the later papers (**7** to **16**), each experiment involved two stages. In the first stage, the specimen (with or without cyclic stress) was corroded by water for a prescribed time, then removed from the water, dried, and oiled. In the second stage, the fatigue limit of the specimen was determined by subjecting it to cyclic stress in air. The decrease in the fatigue limit was used as a measure of the damage resulting from the prior corrosion.

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[1] Metallurgist, and Assistant Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 731.

TABLE I.—CHEMICAL COMPOSITION OF STEELS.

Material	Designation	Carbon, per cent	Manganese, per cent	Silicon, per cent	Chromium, per cent	Nickel, per cent
Carbon steel	AT	0.36	0.55	0.3	0.014	
	JR	0.46	0.65	0.016		
Nickel steel	IW	0.28	0.5	0.19	0.26	3.7
	LQ[a]					
Chromium-nickel steel	AX	0.36	0.48	0.22	1.55	3.26
	AY	0.43	0.60	0.21	0.95	2.16

[a] About the same composition as IW.

TABLE II.—HEAT TREATMENT OF STEELS.

Material	Designation	Heat Treatment					
		Temperature, deg. Fahr.	Time Held, min.	Cooled in	Temperature, deg. Fahr.	Time Held, min.	Cooled in
Carbon steel	AT-W-10	1550	60	Water	1000	120	Furnace
	JR-W-10	1500	60	Water	1000	120	Furnace
Nickel steel[a]	IW-W-10	1450	60	Water	1000	120	Furnace
	LQ-W-10	1450	60	Water	1000	120	Furnace
Chromium-nickel steel	AX-W-10	1550	60	Water	1000	120	Furnace
	AY-W-10	1550	60	Water	1000	120	Furnace

[a] Previously heated to 1675 F. for 60 min., and cooled in air.

TABLE III.—TENSILE PROPERTIES OF STEELS.

Material	Designation	Tensile Strength (TS), psi.	Johnson's Limit, psi.	Proof Stress, psi.	Elastic Limit, psi.	Proportional Limit, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent
Carbon steel	AT-W-10	97 500	71 000	71 500	70 000	70 000	25.5	60.4
	JR-W-10	129 800	100 000	101 400	99 500	92 300	17.5	51.9
Nickel steel	IW-W-10	128 100	114 000	114 600	110 000	94 300	20.6	61.8
	LQ-W-10	133 700	121 300	123 300	121 300	106 400	21.0	63.8
Chromium-nickel steel	AX-W-10	157 700	141 000	142 000	139 000	115 000	17.5	50.7
	AY-W-10	159 000	143 100	144 200	135 200	103 400	15.5	52.0

NOTES.—Usually these values are the average for at least 4 determinations.

Johnson's limit is the stress at that point on the stress-strain diagram at which the tangent is two-thirds that at the origin. Proof stress is the stress that, after release of load, will cause permanent extension of 0.01 per cent. Elastic limit is the highest stress that, after release of load, will cause no appreciable permanent extension. Proportional limit is the highest stress that will not cause appreciable departure from a linear stress-strain relationship. (The smallest divisions of the extensometer scale were 0.0001 in. but estimate could be made to 0.00002 in. The extensometer was of a type that permitted accurate determination of permanent extension.)

Opportunity was not then available for a study of the forms and sizes of the corrosion pits and their correlation with the cyclic stresses applied during corrosion. Later, however, through the courtesy of the Director of the U. S. Naval Engineering Experiment Station, Annapolis, Md. (where the stress-corrosion experiments had been made), and with the cooperation of W. C. Stewart, Metallurgist of the Station, the specimens that had been used were made available for such a study. The results of this examination have been used in two previous papers (**18**, **19**) and in this paper to correlate the forms and sizes of the corrosion pits with the lowering of the fatigue limit and to study the influence of stress on corrosion pitting. The first paper considered the corrosion pitting of steels in well water; the second paper considered the corrosion pitting of aluminum bronze and monel metal in well water. The present paper deals with the pitting of steels under various conditions of corrosion with and without stress, and studies the factors involved in the resultant lowering of the fatigue limit.

I. Materials and Method of Investigation

Steels:

The compositions of the steels are given in Table I, details of heat treatment are given in Table II, and tensile properties are given in Table III.

Specimens:

The specimens used in the corrosion experiments were fatigue test specimens of the rotating cantilever type, fully described in a previous paper (1). The specimens were conically tapered so that the maximum stress occurred $\frac{3}{4}$ in. out from the inner fillet, and so that the stress varied only about $1\frac{1}{2}$ per cent over a length of $1\frac{1}{2}$ in.; the diameter at the inner fillet was $\frac{1}{2}$ in. With this specimen, therefore, a comparatively large region could be subjected to simultaneous corrosion and cyclic stress.

The Corrosion Stage:

In the corrosion stage, some specimens were corroded without stress; other specimens were subjected to various cyclic stresses and cycle frequencies. After corrosion for the desired time, the specimen was removed from the water, dried, and oiled. It was then ready for insertion in another machine in order to determine its fatigue limit in air.

The stressless corrosion experiments considered in this paper were made under various corrosion conditions. In some experiments, water was applied in a small stream, which was directed diagonally to the revolving cantilever specimen so that the entire tapered portion was covered by a thin moving layer of water. The size of the water stream, within wide limits, had no apparent effect on the damage, as measured by the lowering of the fatigue limit. The water used in the streams was either well water, distilled water, or Severn River water (Table IV). In some experiments, water was applied to each specimen in the form of a spray. The apparatus used for this purpose was of the type used in salt-spray tests in common use. The water used in the sprays was either a normal salt solution or a chemically softened water obtained from an artesian well at the Naval Academy (Table IV).

The well water was essentially a solution of calcium and magnesium bicarbonates, calcium sulfate, and chlorides. The chloride content is unusually high for a well water. The saline content of the Severn River water varies greatly, but averages about one-sixth that of sea water.

TABLE IV.—COMPOSITION OF WATER, PARTS PER MILLION.

These values are the average for at least 4 determinations

Water	Solids	Alkalinity[a]	Cl	SO_3	CaO	MgO	pH value
Well	540	103	200	18	96	33	7.1
Distilled	20	12	5	none	0.2	0.8	7.1
Severn	10 500	70	5100	620	200	620	8.9
Softened	115	61	6	26	3	0.3	8.3

[a] Alkalinity is expressed in terms of $CaCO_3$.

Determination of the Fatigue Limit and Evaluation of the Damage Caused by the Corrosion:

In the second stage, the fatigue limit of each corroded specimen was determined by a rotating cantilever test. In the early experiments (7), it was necessary to test several similarly corroded specimens in order to determine the fatigue limit corresponding to a prescribed time, stress, and cycle frequency in the corrosion stage. These specimens were subjected to different cyclic stresses, and determination was made of the numbers of cycles necessary to cause fatigue failure. A stress-cycle curve could then be plotted and an estimate could be made of the fatigue limit.

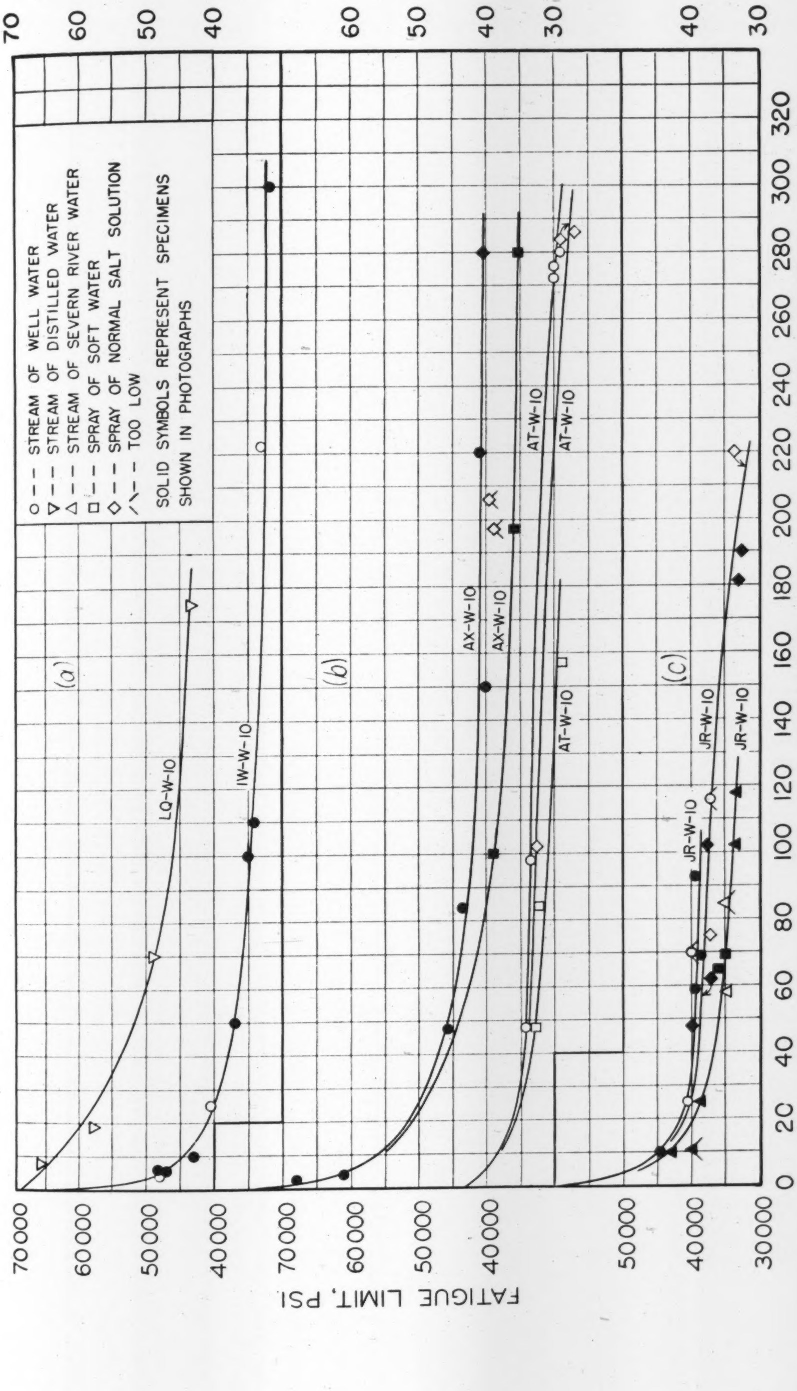

FIG. 1.—The Lowering of the Fatigue Limit Due to Stressless Corrosion Under Various Conditions.

Later, however, it was generally found possible to make a satisfactory estimate of the fatigue limit by use of only one specimen for a prescribed stress, cycle frequency and corrosion condition. That was accomplished by selecting a stress that would cause failure in not much less than a million cycles. The fatigue limit was then estimated by extrapolation to ten million cycles (**9, 17**).[3] The lowering of the fatigue limit due to the corrosion was used as a measure of the damage.

The Method of Investigation of the Forms and Sizes of the Corrosion Pits:

The outer surfaces of the specimens were examined visually and photographed at low magnification (about 4). The pits were also viewed in longitudinal sections cut approximately through the axes of the specimens. Typical photographs, originally at 100 magnifications, have been reduced to 50 magnifications. The photographs of the surfaces were made after removal of enough of the general rust layer to reveal the corrosion pits, without removal of the corrosion products from the pits.

To insure a correct sectional view of the pits, the polishing of the sections was done in such a way as to minimize removal of the corrosion products. The specimens were prepared by dry polishing; at an early stage in the polishing, the specimen was mounted in Wood's metal. Each section was ground on a series of emery papers of increasing fineness, and finally with Aloxite 400 paper. It was then rubbed on 00 emery paper (previously somewhat worn) lubricated with graphite from a brush block. It was next polished by means of a revolving disk covered with a good grade of chamois skin, with smooth (hair) side outward. A small quantity of No. 600 emery powder was used on this wheel. The specimen was then transferred to an automatic polishing machine, and polished on a disk covered with chamois skin. The polishing powder used in this final stage was magnesium oxide. Practice is necessary to determine the proper length of time in each polishing stage and the proper amount of polishing powder.

II. The Lowering of the Fatigue Limit of Steels Due to Stressless Corrosion Under Various Conditions

The influence of corrosion on the fatigue limit may be represented by diagrams of the type shown in Fig. 1. The abscissa represents duration of corrosion and the ordinate represents the resultant fatigue limit. Most of the experimental points on which the curves are based were obtained with the same specimens that have been used in the investigation of the forms and sizes of corrosion pits. (Section III.)

In Fig. 1 are curves representing the lowering of the fatigue limit of various steels due to stressless corrosion under various conditions. Each of these curves descends rapidly at first, but the slope decreases until the curve becomes nearly horizontal. Curves of this type have been called retarded-damage curves (**18**). Curves of a very different type, called "accelerated damage" curves, have been obtained with stainless steel (**10, 11, 14, 15**), nickel (**14**), aluminum bronze (**14, 19**), and monel metal (**19**). An accelerated-damage curve is nearly horizontal at first and remains so throughout a long period of corrosion. Eventually, however, the slope begins to increase at an increasing rate and finally becomes steep.

In Fig. 1(*c*) are curves representing the lowering of the fatigue limit of a carbon

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[3] The loss of accuracy owing to the use of only one specimen was slight, and was greatly outweighed by the increase in number of stress-corrosion experiments thus made possible.

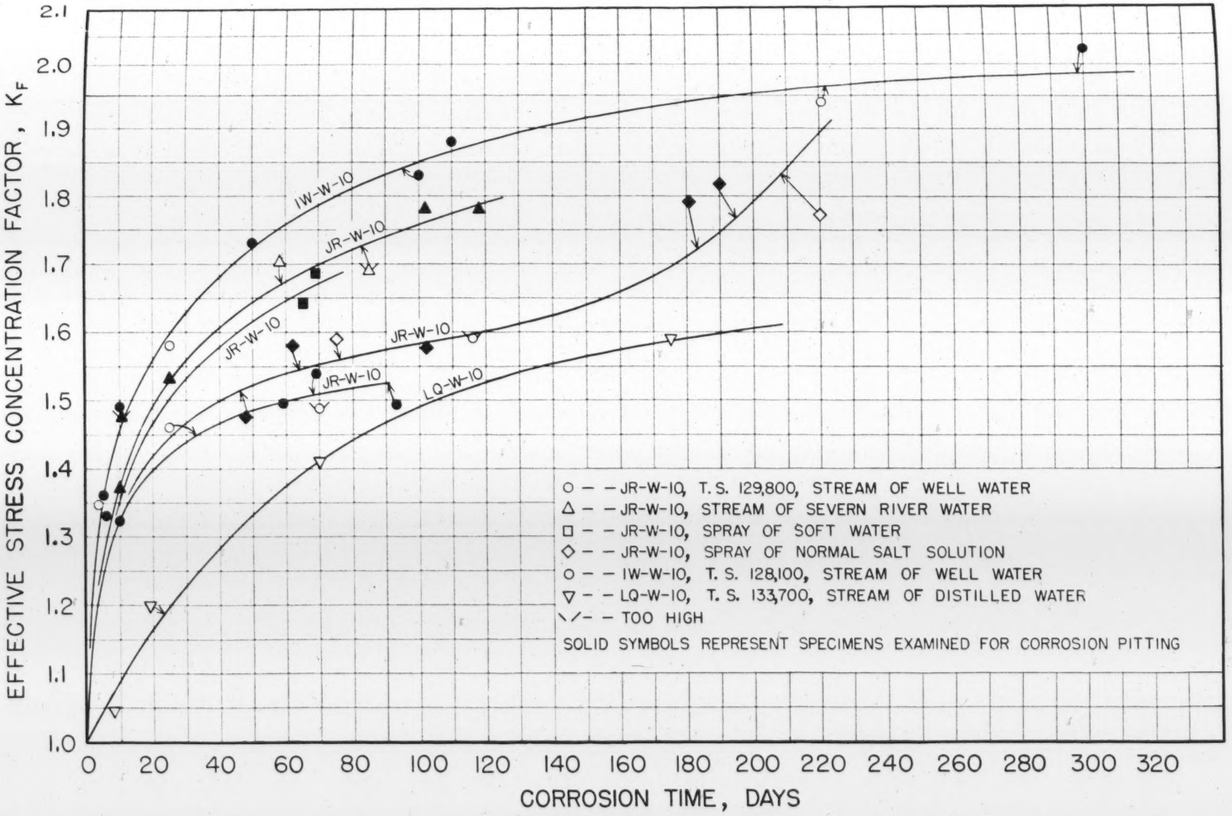


FIG. 2.—The Effective Stress Concentration Due to Stressless Corrosion of Various Steels Under Various Conditions.

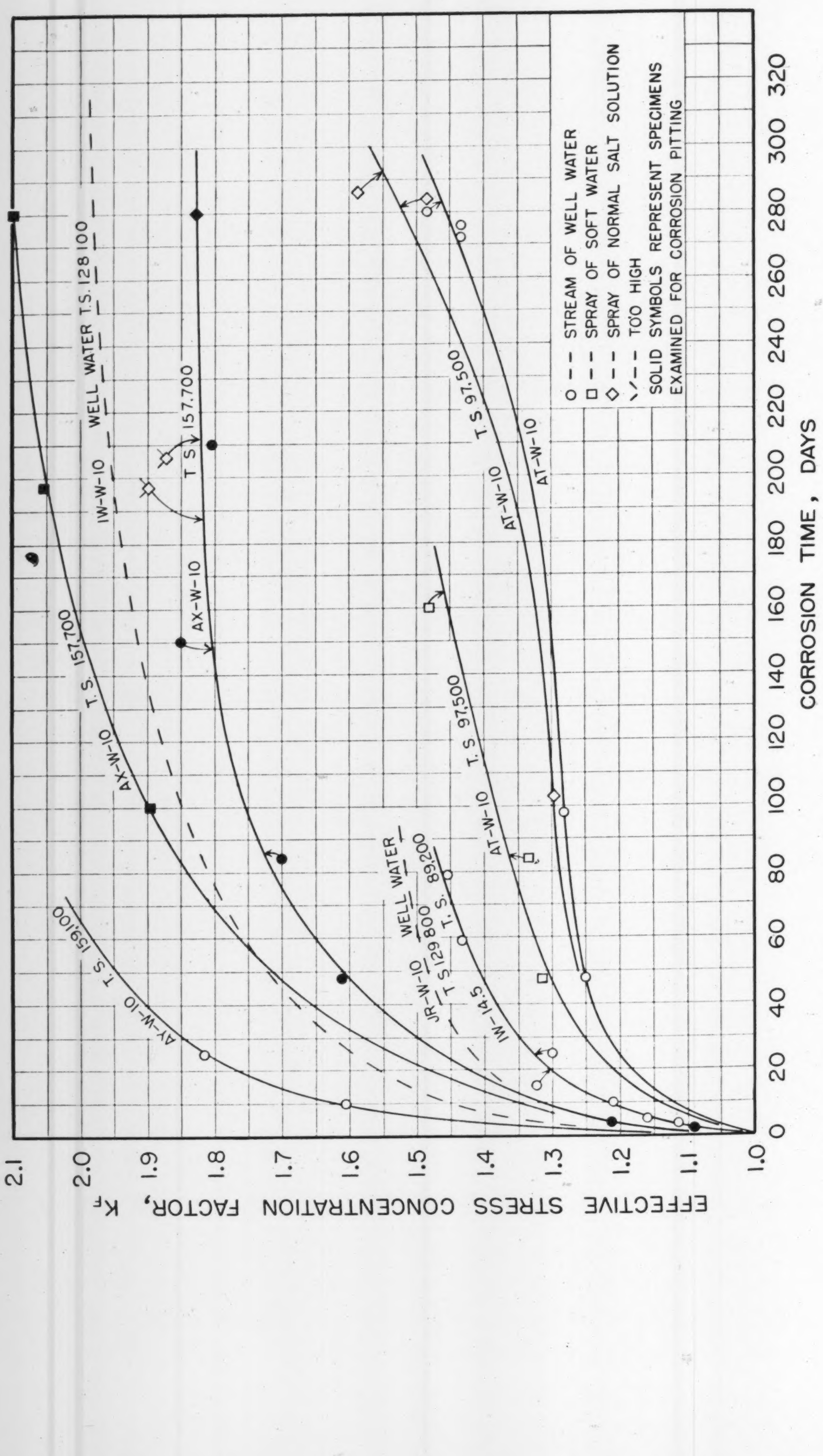


Fig. 3.—The Effective Stress Concentration Due to Stressless Corrosion of Various Steels Under Various Conditions.

steel due to stressless corrosion under four different conditions. The highest of the three curves represents the results of corrosion in a stream of well water; the lowest represents corrosion in a stream of Severn River water. Only slightly above the lowest curve in the diagram are the points representing corrosion in a spray of chemically softened artesian well water (Table IV). Reversal of curvature is found in the curve representing corrosion in a spray of normal salt solution. Throughout the first 60 days of corrosion, this curve differs little from the curve representing corrosion in a stream of well water; the curvature then is reversed, and the curve descends at an increasing rate. The curve representing corrosion in well water, if extended far enough, possibly would have a similar reversal.

The positions of the three curves in Fig. 1(*c*) are not in the order of the percentages of chlorides in the water (Table IV). Of the two curves obtained with streams of water, the one obtained with the brackish Severn River water is below the one obtained with the well water. The spray of soft water, however, lowered the fatigue limit more than did the spray of normal salt solution (containing 5.85 per cent NaCl). It was expected that a curve obtained with a spray of soft water would differ little from the curve obtained with a stream of well water and would be above the curve representing corrosion in a spray of normal salt solution. That the relationship found is not exceptional, however, is indicated by the curves obtained with a different carbon steel and with an alloy steel (Fig. 1 (*b*)).

The relative positions of the curves obtained with carbon steel AT-W-10 (Fig. 1 (*b*)) are the same as in the diagram obtained with carbon steel JR-W-10 (Fig. 1 (*c*)), at least throughout the first 160 days of corrosion. The curve obtained with a spray of soft water is below the curve obtained with a spray of normal salt solution. The curve obtained with the spray of normal salt solution, moreover, reverses its curvature and descends at an increasing rate, as it does in Fig. 1 (*c*). The curve obtained with a stream of well water has a similar reversal and differs little from the curve obtained with the spray of normal salt solution. The curves representing chromium-nickel steel AX-W-10, (Fig. 1(*b*)) are in the same relative positions as the corresponding curves obtained with the carbon steels (Fig. 1 (*b*) and (*c*)). The curve and experimental points obtained with this steel in a stream of well water and in a spray of normal salt solution, however, give no evidence of a reversal of curvature similar to that shown in the corresponding curves for the two carbon steels, AT-W-10 and JR-W-10.

In Fig. 1 (*a*) are curves obtained by stressless corrosion of two nickel steels in streams of well water and distilled water. Because of the smaller content of salt and other solids, the rate of lowering of the fatigue limit was much less in the distilled water than in the well water. In a previous paper (18), these curves obtained with well water are correlated with the form, size, and distribution of the corrosion pits.

From the curves in Fig. 1 have been derived curves of another type shown in Figs. 2 and 3. In Section IV, these curves are correlated with the form, size, and distribution of the corrosion pits.

III. PITTING OF STEELS BY STRESSLESS CORROSION UNDER VARIOUS CONDITIONS

Pitting Caused by Stressless Corrosion in a Stream of Well Water:

Figures 4 and 5 show surface views and Figs. 6 and 7 show longitudinal sections of specimens after stressless corrosion in a stream of well water. In the surface views, the vertical direction represents the longitudinal direction on the surface of a specimen. Each surface view includes practically the entire width of a specimen. As the photograph shows a curved surface, the view is oblique

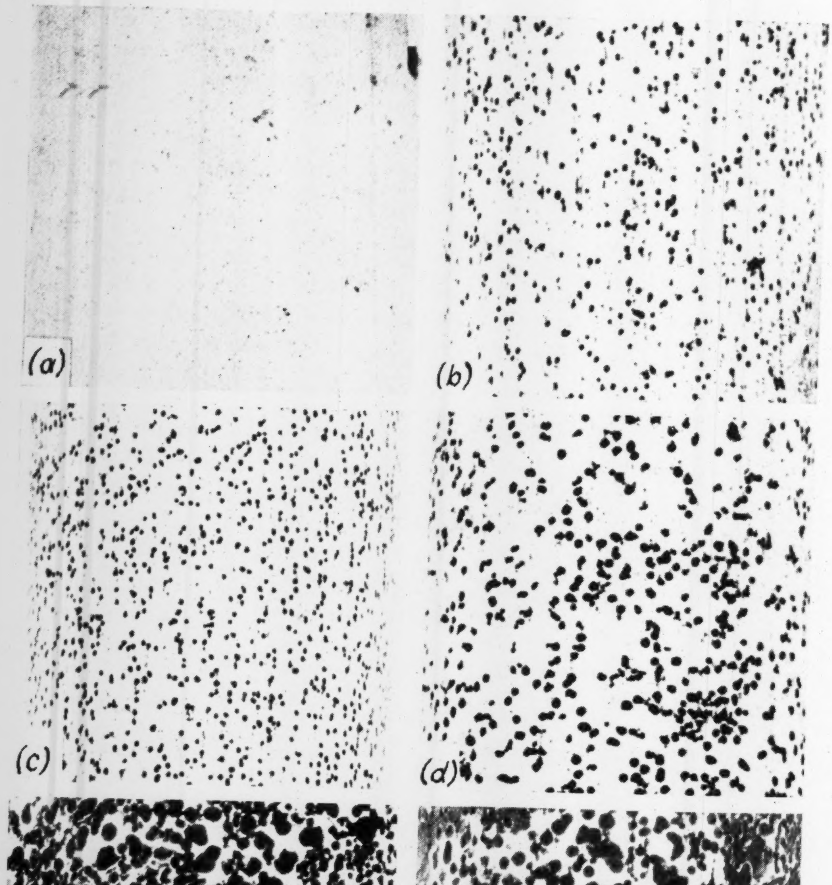


FIG. 4.—Stressless Corrosion, Stream of Well Water, Outer Surfaces (× 4).

(a) 2 days, AX-W-10
(b) 4.7 days, IW-W-10
(c) 5.8 days, IW-W-10
(d) 10 days, JR-W-10
(e) 50 days, IW-W-10
(f) 59 days, JR-W-10

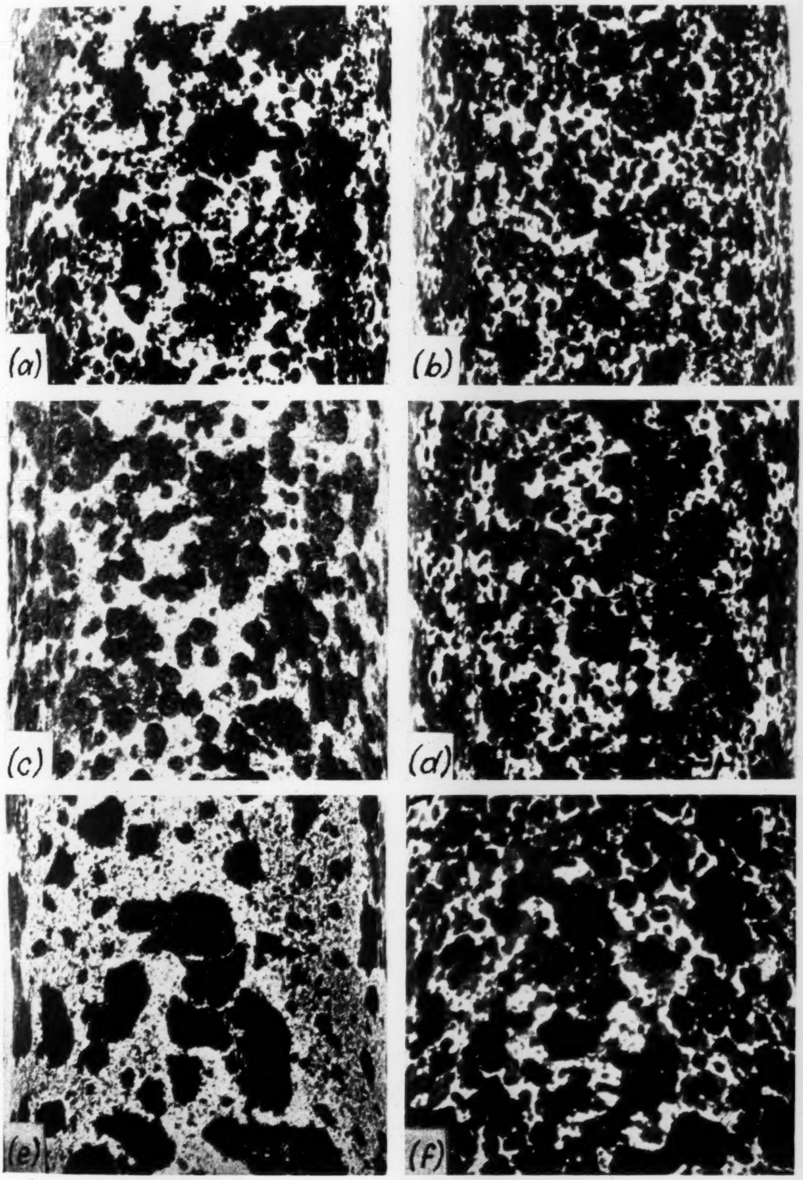


FIG. 5.—Stressless Corrosion, Stream of Well Water, Outer Surfaces (× 4).

(a) 69 days, JR-W-10
(b) 93 days, JR-W-10
(c) 100 days, IW-W-10
(d) 150 days, AX-W-10
(e) 220 days, AX-W-10
(f) 300 days, IW-W-10

except at the center line, and the obliquity increases from this line toward the right and left edges. This should be kept in mind in studying the forms, sizes, and distribution of the corrosion pits.

In the sectional views (Figs. 6 and 7), the Wood's metal appears at the left of each photograph. Although part of the corrosion products has been detached from some of the pits during examination of the outer surface, enough of these products remains to ensure that the photographs give the true forms of the pits. It should be kept in mind that these sections do not pass through the centers of all the visible corrosion pits. An attempt has been made, however, to make each section pass approximately through the center of at least one pit. The micrographs shown have been

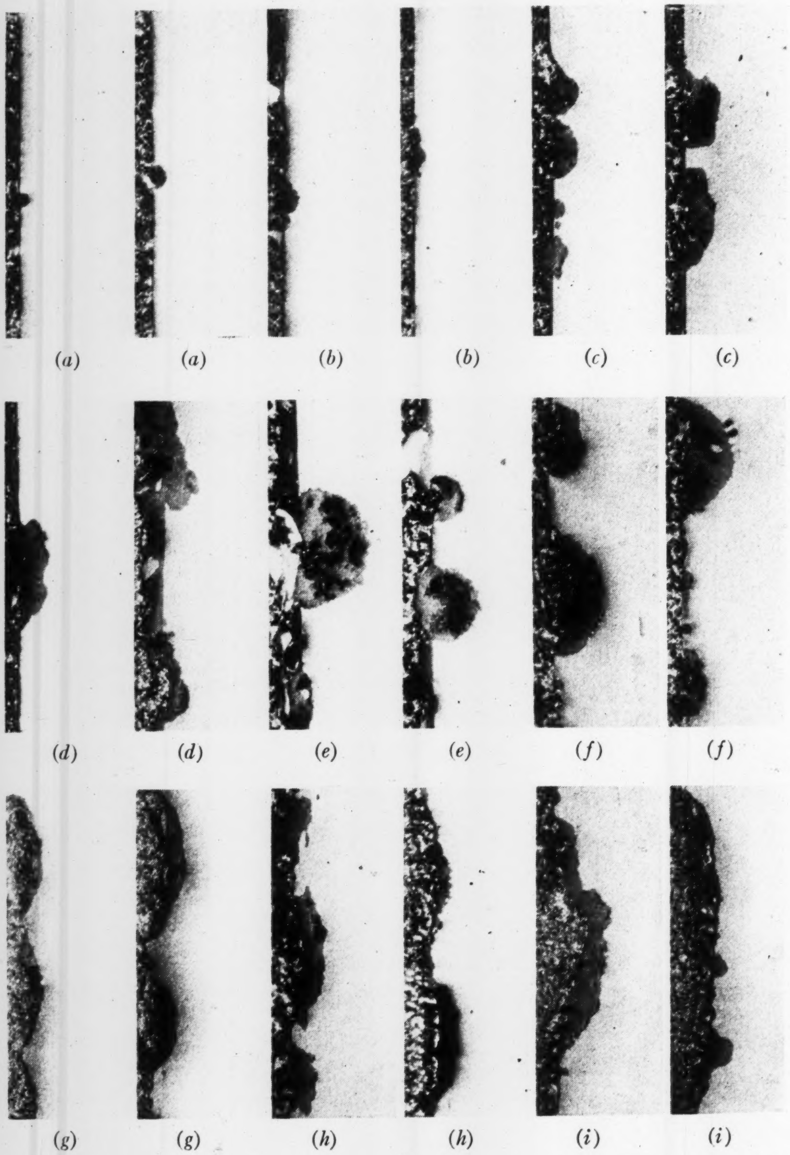


Fig. 6.—Stressless Corrosion, Stream of Well Water, Longitudinal Sections (× 50).

(a) 2 days, AX-W-10
(b) 4 days, AX-W-10
(c) 4.7 days, IW-W-10
(d) 10 days, IW-W-10
(e) 47 days, AX-W-10
(f) 50 days, IW-W-10
(g) 59 days, JR-W-10
(h) 69 days, JR-W-10
(i) 84 days, AX-W-10

selected from several (often five or more) of each section, and each section is represented by two or more separate micrographs or by a larger continuous micrograph.

During the first few days of corrosion the pits appear on the surfaces as round spots (Fig. 4 (*a*) to (*d*)). The size of the pits increases with the duration of corrosion and some adjacent pits soon

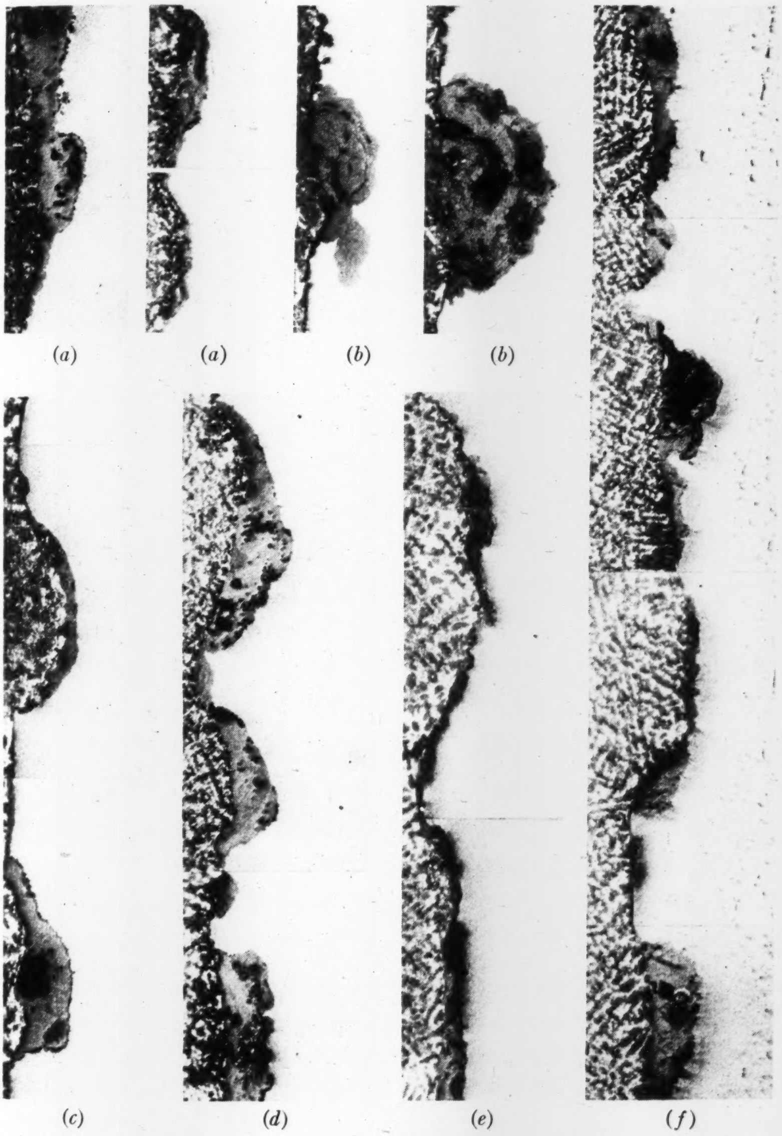

(*a*) (*a*) (*b*) (*b*)
(*c*) (*d*) (*e*) (*f*)

FIG. 7.—Stressless Corrosion, Stream of Well Water, Longitudinal Sections (× 50).
(*a*) 93 days, JR-W-10
(*b*) 100 days, IW-W-10
(*c*) 110 days, IW-W-10
(*d*) 150 days, AX-W-10
(*e*) 220 days, AX-W-10
(*f*) 300 days, IW-W-10

merge. After 50 and 59 days (Fig. 4 (*e*) and (*f*)), the pits have increased greatly in size, and occupy a large proportion of the surface. The pitting now appears in two shades, depending on the depth of pitting. The darker shade represents round pits, either on the surface or within the boundaries of larger pits of lighter shade. Several dark, round spots can be seen within many of the lighter areas; these spots are larger than those found after corrosion for only 10 days (Fig. 4 (*d*)). The pits evidently have grown by lateral extension, involving the merging of many of the originally round pits.

With further increase in the corrosion time, as shown in Fig. 5 (69 to 300 days), the pits continued to grow, but much less rapidly than during the first 50 days. Although the growth was largely by lateral extension, involving the merging of originally round pits, a few round pits are still visible both on the surface and within the larger, lighter pits. These relatively deep, round pits are conspicuous in Fig. 5 (*a*), (*d*), and (*f*). They are generally larger than the round pits found after 50 and 59 days (Fig. 4 (*e*) and (*f*)).

Sections through many of the pits that appear roughly circular in the surface views (Figs. 4 and 5) appear roughly semicircular in Figs. 6 and 7. This evidence, supplemented by evidence found in other views shown in a previous paper (**18**), indicates that a large proportion of the pits caused by stressless corrosion in this water are roughly hemispherical. Some roughly hemispherical pits are found even after other pits in the same specimen have extended laterally and merged (compare Figs. 4 (*e*) and 5 (*c*) with Figs. 6 (*e*) and (*f*) and 7 (*b*)). Even in the early stages of corrosion, however, some of the pits are saucerlike rather than hemispherical; this is illustrated in Fig. 6 (*b*), (*c*), and (*d*). In later stages, owing to lateral extension of the pits, the saucerlike form is common. Even after the merging of the pits, as illustrated in Fig. 6 (*g*) and (*h*) and Fig. 7 (*a*), (*e*), and (*f*), the bottoms of the merged pits generally consist of numerous saucers. As will be shown later, the rapidity of the change from hemispherical to saucerlike form depends somewhat on the composition of the steel.

After corrosion for 50 days or more, small salients are visible, projecting into the metal from many of the corrosion pits (Figs. 6 and 7). Even after 10 days' corrosion, smaller projections are visible at higher magnification. These rootlike projections probably are due to the influence of oxygen-concentration cells.

The size of the pits increases greatly during the first few days of corrosion. This is seen both in the surface views (Fig. 4 (*a*) to (*d*)) and in the longitudinal sections (Fig. 6 (*a*) to (*d*)). With increase in the corrosion time beyond about 10 days, the increase in (relative) size of the corrosion pits is slower; nevertheless the growth is considerable between about 50 and 100 days. (Compare Fig. 6 (*e*) and (*f*) with Fig. 7 (*b*).) Beyond 100 days, the depth evidently increases little but the breadth increases, largely by the merging of adjacent pits.

Pitting Caused by Stressless Corrosion in a Stream of Distilled Water:

The distilled water used was obtained from the condenser of a steam boiler. This water was less pure than laboratory distilled water. The total solids, however, were much less than in the other waters used (Table IV). Longitudinal sections through the four specimens that were subjected to stressless corrosion in distilled water are shown in Figs. 8 and 9. During the first few days of corrosion, many of the pits evidently are roughly hemispherical like the pits caused by stressless corrosion in well water. With increase in the corrosion time, however, the pits become saucerlike and then spread further along the surface. The tendency of the pits to spread is greater in distilled water than in well water.

The growth of the hemispherical pits, moreover, is slower in distilled water than in well water. After 8 and 19 days corrosion in distilled water (Fig. 8 (*a*) and (*b*)) the pits appear smaller than after 4.7 days in well water (Fig. 6 (*c*)). After 175 days corrosion in distilled water (Fig. 9) the pits appear smaller than those found after 100 and 110 days in well water (Fig. 7 (*b*) and (*c*)).

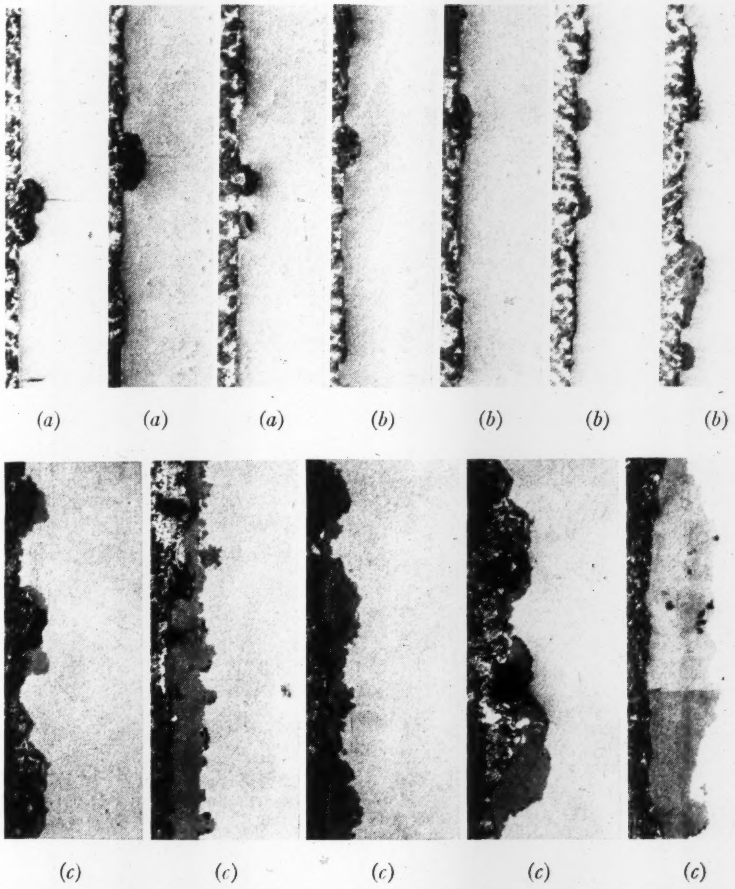


Fig. 8.—Stressless Corrosion, LQ-W-10, Stream of Distilled Water, Longitudinal Sections (× 50).
(*a*) 8 days (*b*) 19 days (*c*) 71 days

Pitting Caused by Stressless Corrosion in a Stream of Severn River Water:

Surface views of specimens after stressless corrosion in a stream of Severn River water are shown in Fig. 10. All but one of the specimens here shown are carbon steels. No views are available of specimens after corrosion for less than 10 days.

Figure 10 (*a*) shows a surface view of a specimen of carbon steel JR-W-10 after corrosion for 10 days. The pits vary greatly in size. The smaller pits are round, like pits caused by stressless corrosion for a few days in well water (Fig. 4). Many of the pits, however, are broad and irregular; within these broad areas are a few indistinct dark round

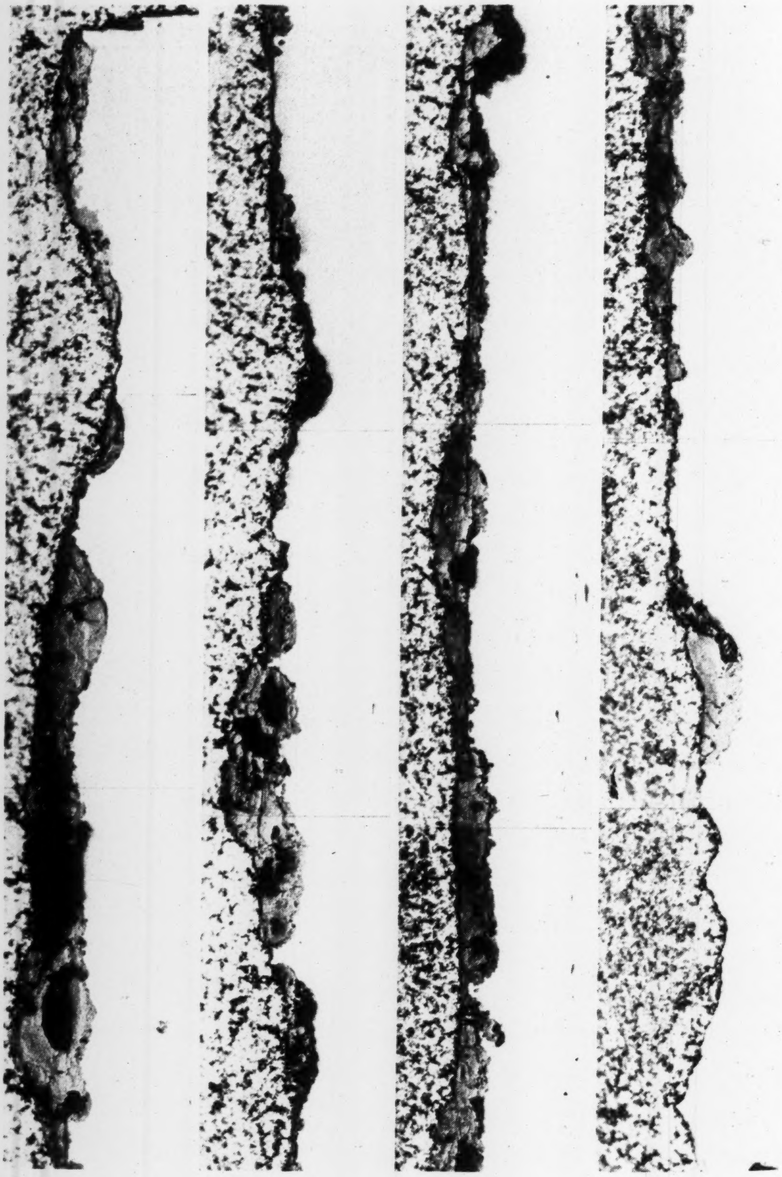


Fig. 9.—Stressless Corrosion, LQ-W-10, Stream of Distilled Water, 175 days, Continuous Longitudinal Section (× 50).

spots, probably representing relatively deep rounded pits. Another specimen of carbon steel corroded for about the same length of time is shown in Fig. 10 (*c*). In this specimen, the pits are fewer than in Fig. 10 (*a*) and many of them have extended further along the surface. A few dark, round spots are visible. A specimen of nickel steel after corrosion for 10 days is shown in Fig.

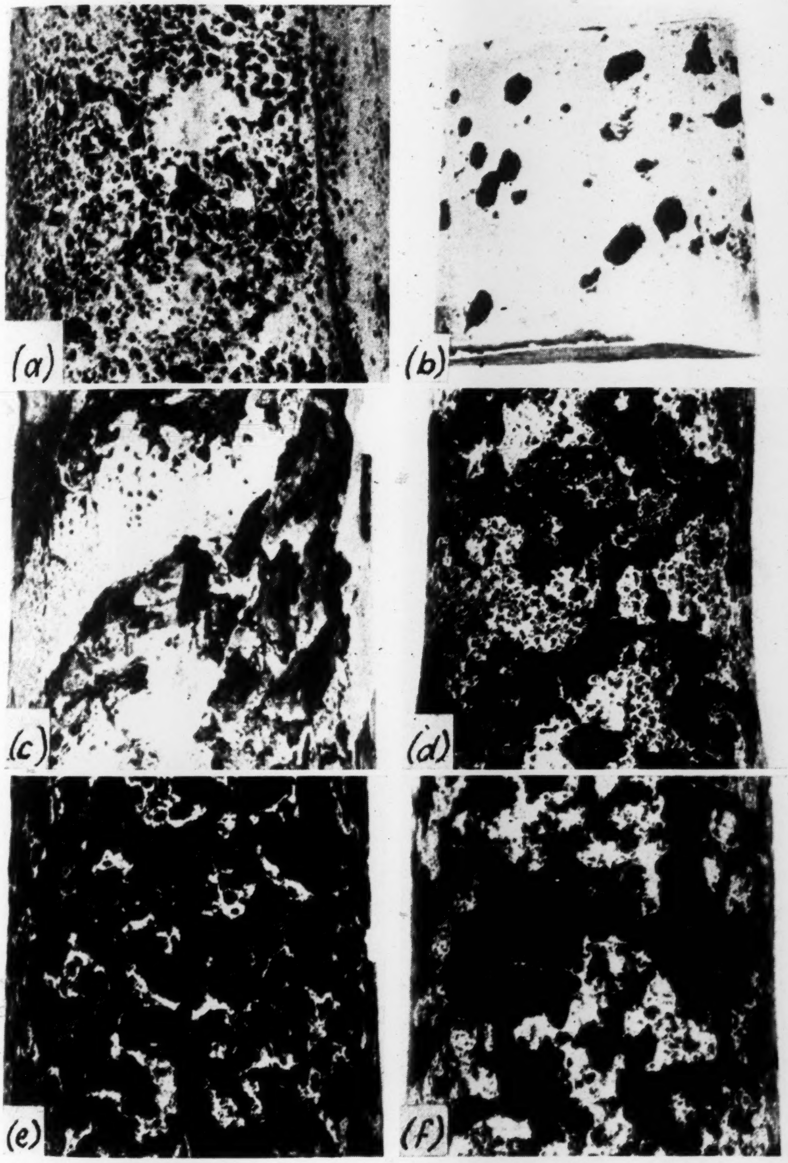


FIG. 10. Stressless Corrosion, Stream of Severn River Water, Outer Surfaces (× 4).

(*a*) 10 days, JR-W-10
(*b*) 10 days, IW-W-10
(*c*) 10.8 days, JR-W-10
(*d*) 25 days, JR-W-10
(*e*) 102 days, JR-W 10
(*f*) 118 days, JR-W-10

10 (*b*). The pits are fewer and the dark round spots are more distinct in this specimen than in the specimens of carbon steel (Fig. 10 (*a*) and (*c*)). This difference may be due partly to the difference in chemical composition. The number of pits per unit area, however, may vary greatly owing to uncontrollable variables under apparently constant corrosion conditions (**18**).

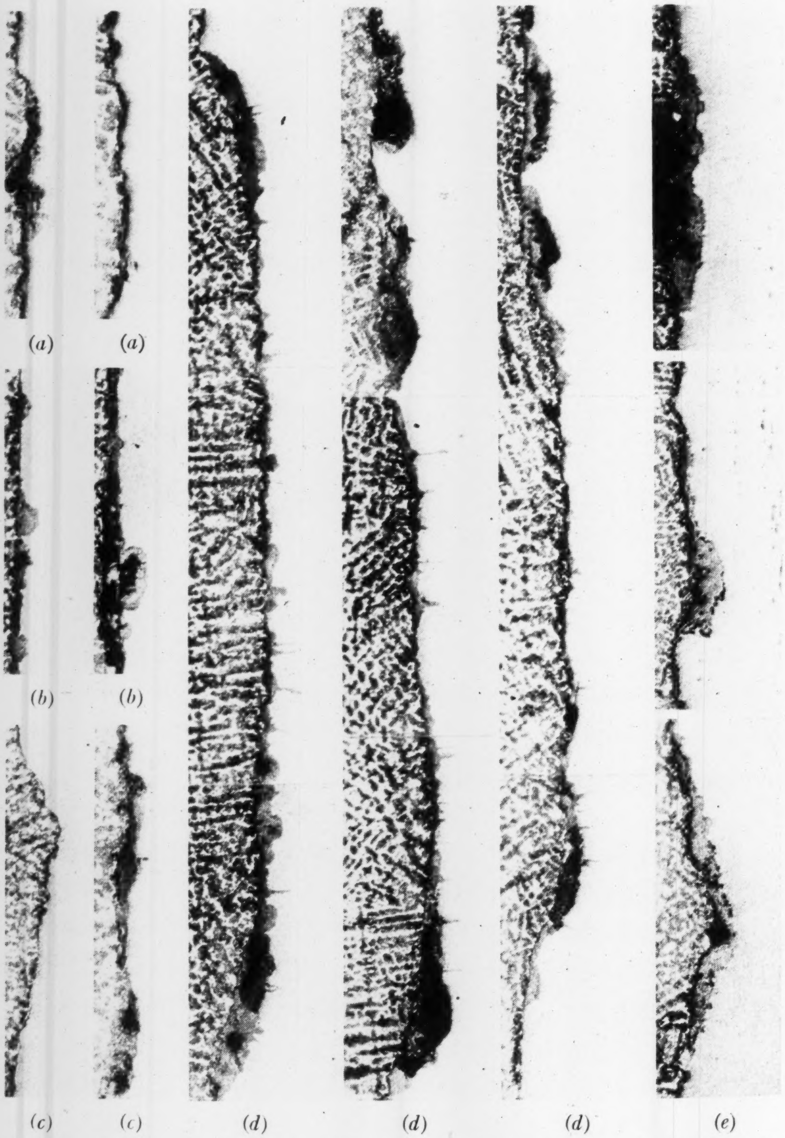


FIG. 11.—Stressless Corrosion, Stream of Severn River Water, Longitudinal Sections (× 50).

(*a*) 10 days, JR-W-10
(*b*) 10.8 days, JR-W-10
(*c*) 25 days, JR-W-10
(*d*) 102 days, JR-W-10
(*e*) 118 days, JR-W-10

With increase in corrosion time (Fig. 10 (*d*), (*e*), and (*f*)) the pits have con-

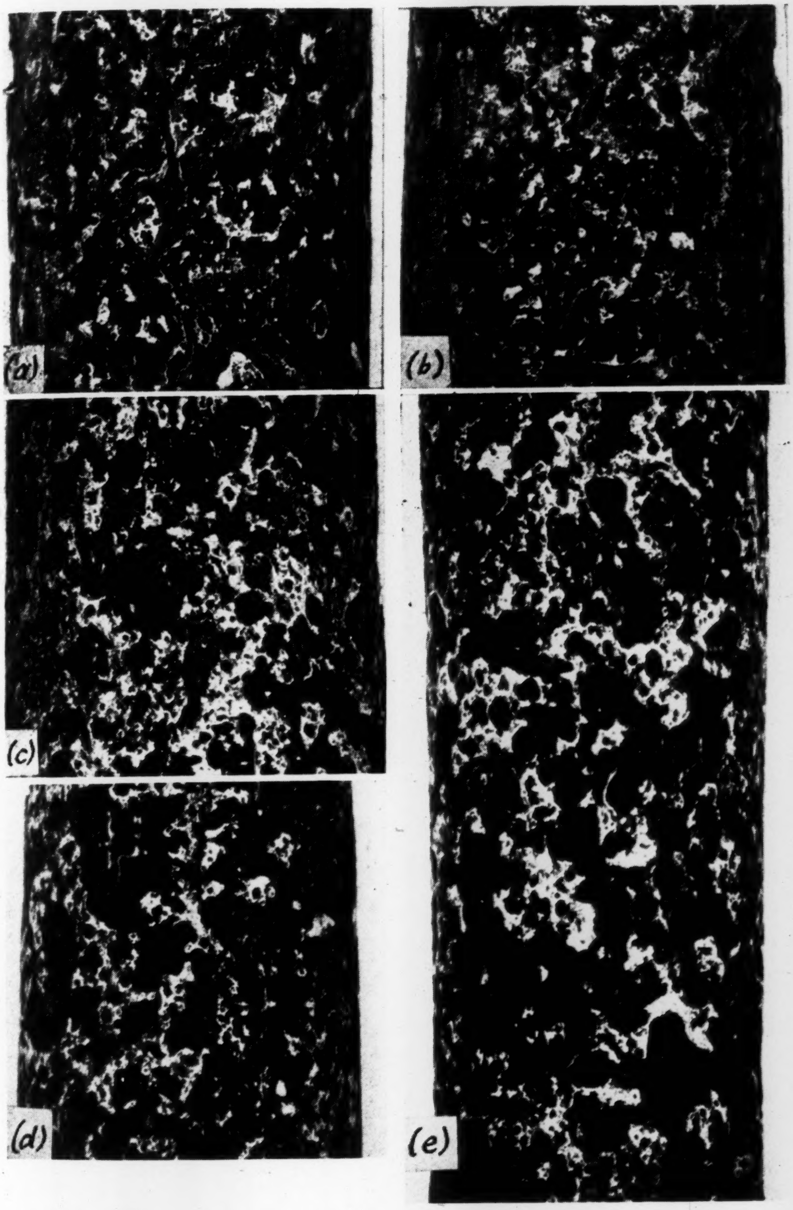
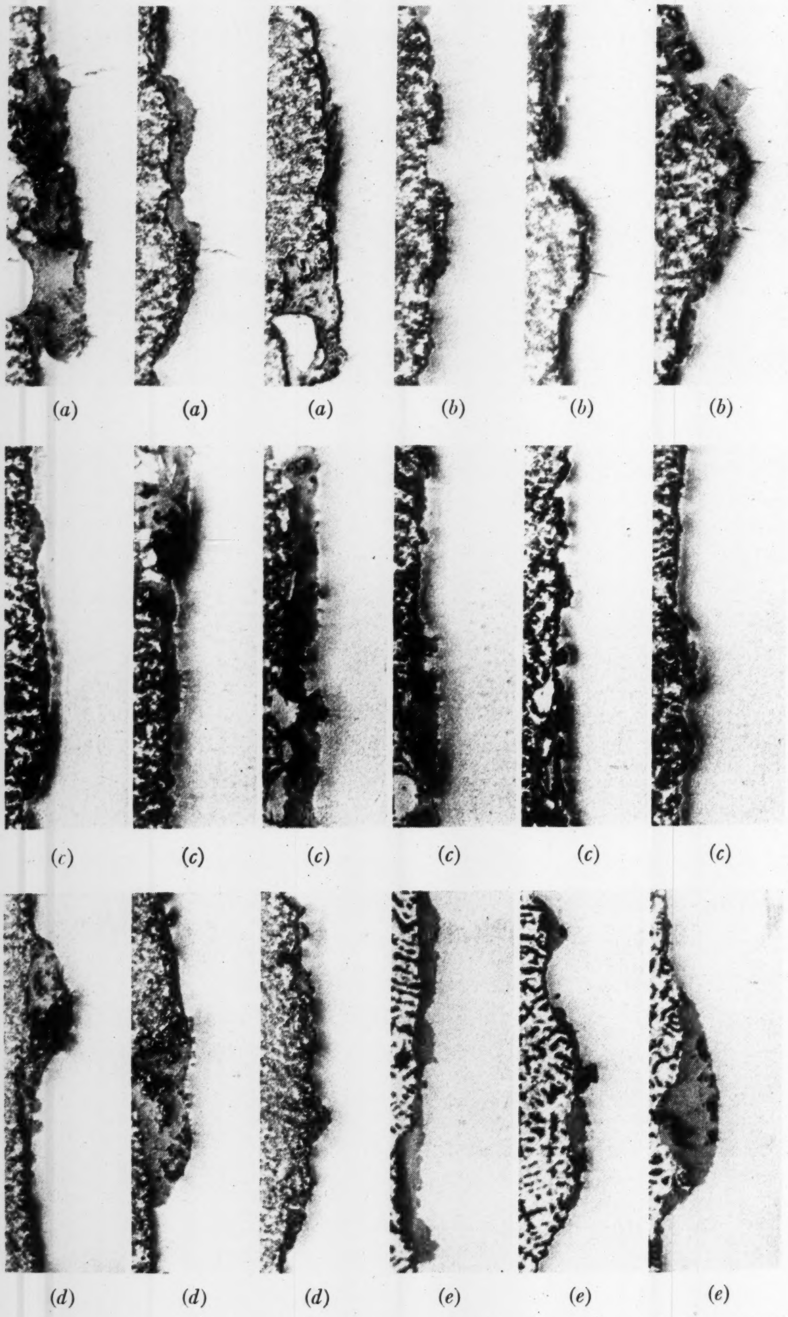


FIG. 12.—Stressless Corrosion, Spray of Softened Water, Outer Surfaces (× 4).

(a) 65 days, JR-W-10
(b) 69 days, JR-W-10
(c) 100 days, AX-W-10
(d) 197 days, AX-W-10
(e) 280 days, AX-W-10

tinued to extend along the surface. A few dark round spots can still be seen. The pits evidently tend to be fewer and to spread more, and the dark round spots thus tend to be much less prominent, after corrosion in Severn River


(a)
(a)
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(c)
(c)
(c)
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(c)
(d)
(d)
(d)
(e)
(e)
(e)

FIG. 13.—Stressless Corrosion, Spray of Softened Water, Longitudinal Sections (× 50).

(a) 65 days, JR-W-10
(b) 69 days, JR-W-10
(c) 100 days, AX-W-10
(d) 197 days, AX-W-10
(e) 280 days, AX-W-10

water than after corrosion in well water (Figs. 4 and 5).

These conclusions are confirmed by comparison of the sectional views in Fig. 11 with those in Figs. 6 and 7. The roughly semicircular pits, prominent even after long corrosion in well water, are very scarce after corrosion in Severn River water. The tendency of the pitting to spread along the surface evidently is much greater in Severn River water than in well water. After corrosion for 102 and 118 days in Severn River water, however, numerous small sharp projections extend inward from the broad corrosion pits (Fig. 11 (*d*) and (*e*)). As the specimens were corroded without stress, these projections probably have a rootlike form. The stress concentration due to such projections is much less than that due to the sharp transverse crevices and fissures that may form under the influence of simultaneous corrosion and cyclic stress.

As shown in Fig. 1 (*c*), the lowering of the fatigue limit is considerably greater during corrosion in Severn River water than during corrosion in well water. The causes of this are discussed in Section IV.

Pitting Caused by Stressless Corrosion in a Spray of Softened Water:

Surface views after stressless corrosion in a spray of softened water (Table IV) are shown in Fig. 12 and longitudinal sections are shown in Fig. 13. The first two views in each of these figures show the effects of corrosion of a carbon steel; the other three views show the effects of corrosion of a chromium-nickel steel. As the specimens were stationary and nearly vertical during corrosion, the thin layer of water on the surface tended to flow longitudinally. The irregular erosion shown in Fig. 12 (*a*) and (*b*), however, indicates that the water flow on the carbon steel specimens became irregular as the surface became roughened. The chromium-nickel steel specimens (Fig. 12 (*c*), (*d*), and (*e*)), however, show little evidence of irregular flow but are more like specimens that have been pitted by a stream of well water (Figs. 4 and 5). The tendency of the pits to spread over the surface of carbon steel evidently is greater in a spray of softened water (Fig. 12 (*a*) and (*b*)) than in a stream of well water (Figs. 4 (*f*) and 5 (*a*)). With chromium-nickel steel AX-W-10, however, there was very little difference in the spreading of the pits under the two corrosion conditions. The evidence thus indicates that the tendency of the corrosion pits to spread over the surface is greater for carbon steel than for chromium-nickel steel, and that this difference between the two steels is especially prominent in a spray of softened water.

The sectional views of the carbon steel (Fig. 13 (*a*) and (*b*)) show broad saucerlike pits. The breadth of these pits is greater than that of the pits in specimens of the same steel after corrosion for about the same lengths of time in a stream of well water. Projections from the rounded pits differ little for these two corrosion conditions but are fewer and less prominent than after corrosion in Severn River water (Fig. 11). As shown in Fig. 1, the lowering of the fatigue limit was more rapid in a spray of softened water than in a stream of well water. Reasons for this difference are given in Section IV.

Pitting Caused by Stressless Corrosion in a Spray of Normal Salt Solution:

Surface views after corrosion in a spray of normal salt solution are shown in Fig. 14 and longitudinal sections are shown in Fig. 15. One view in each figure shows effects of corrosion of chromium-nickel steel AX-W-10; the other views show effects of corrosion of carbon steel JR-W-10.

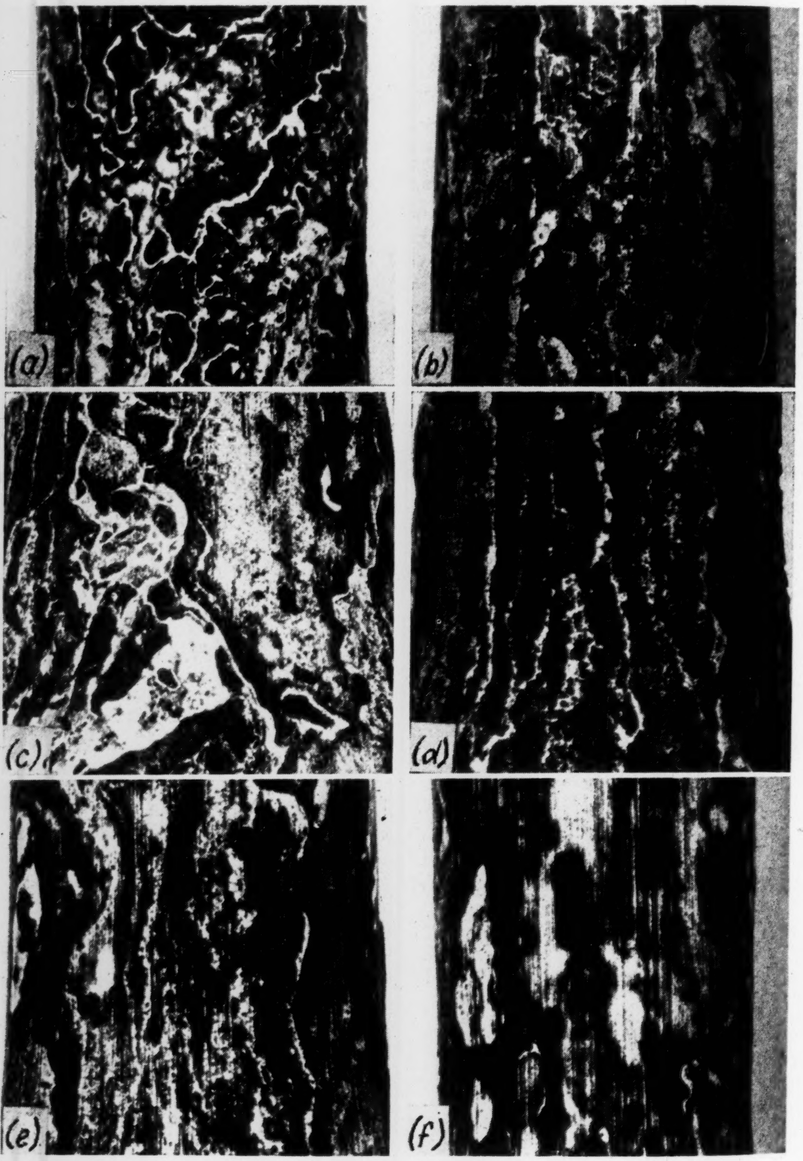

Fig. 14.—Stressless Corrosion, Spray of Salt Solution, Outer Surfaces (× 4).
(a) 48 days, JR-W-10
(b) 62 days, JR-W-10
(c) 102 days, JR-W-10
(d) 181 days, JR-W-10
(e) 190 days, JR-W-10
(f) 280 days, AX-W-10

The surface views show irregular erosion under the influence of the generally longitudinal flow of the corrosive liquid. Dark round spots are visible in all the surface views of the carbon steel (Fig. 14) but none is visible in the view of the

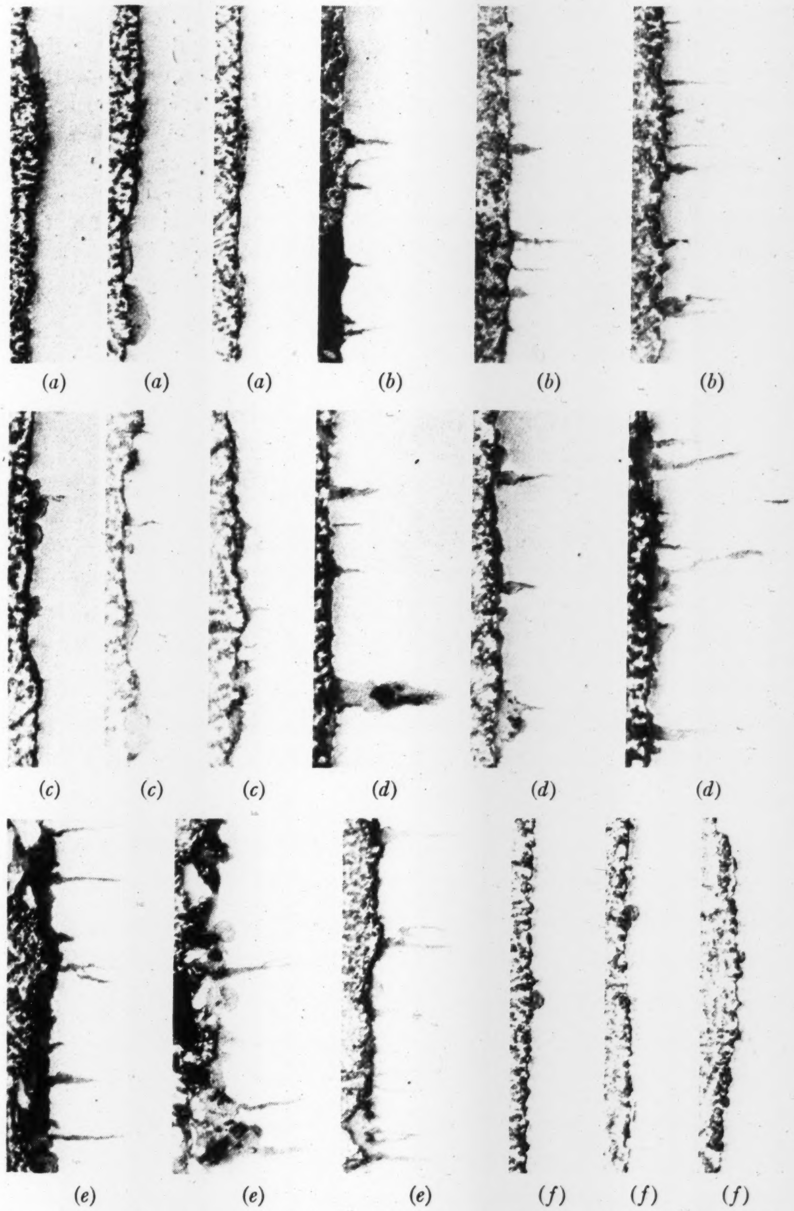

FIG. 15.—Stressless Corrosion, Spray of Salt Solution, Longitudinal Sections (× 50).

(a) 48 days, JR-W-10
(b) 62 days, JR-W-10
(c) 102 days, JR-W-10
(d) 181 days, JR-W-10
(e) 190 days, JR-W-10
(f) 280 days, AX-W-10

chromium-nickel steel (*f*). These round spots are views of rootlike pits such as those visible in the sectional views (Fig. 15). After corrosion for 48 days no rootlike pits are found in the sectional view (Fig. 15 (*a*)) although such pits apparently are revealed by the corresponding surface view (Fig. 14 (*a*)). Numerous rootlike projections, however, are visible in all the other sectional views of the carbon steel (Fig. 15). The size of these projections evidently tends to increase with duration of corrosion. The specimen of chromium-nickel steel (*f*) shows no evidence of rootlike projections; the sectional view thus confirms the impression given by the surface view (Fig. 14 (*f*)).

In spite of the rootlike projections, however, the lowering of the fatigue limit was less rapid in the spray of normal salt solution than in the spray of softened water (Fig. 1). Reasons for this relationship are given in the next section.

IV. STRESS CONCENTRATION CAUSED BY CORROSION PITS IN STEELS

The Effective Stress Concentration Factor:

The lowering of the fatigue limit caused by corrosion pits as by any other notch must be attributed to stress concentration. Because of stress concentration, the actual stress around a notch may be above the fatigue limit when the nominal stress (estimated from the applied load and the dimensions of the specimen) is much less. Any attempt to correlate the forms and sizes of corrosion pits with the lowering of the fatigue limit must be based on a study of the factors affecting the stress concentration.

The effective stress-concentration factor may be denoted by the ratio of the fatigue limit of an unnotched specimen to the fatigue limit of the notched specimen. This ratio, K_F, depends not only on the theoretical stress concentration factor K but also on the size of the notch and on the notch sensitivity of the metal. The theoretical stress-concentration factor depends only on the form of the specimen and notch, not on the size. The notch sensitivity of the metal depends on the chemical composition and on the mechanical properties. The larger the size of the notch and the greater the notch sensitivity, the nearer does the effective stress concentration factor K_F approach the theoretical stress concentration factor K.

The Theoretical Stress Concentration Due to Corrosion Pits:

Although the theoretical stress concentration factor due to corrosion pits cannot be estimated exactly, an approximate estimate may be made of the minimum value and of the probable range of significant values. As the pits during the first few days of stressless corrosion in a stream of well water or distilled water are approximately hemispherical, the simplest approach to a study of the theoretical stress concentration is made by assuming that all the pits are modifications of smooth hemispherical pits.

The theoretical stress concentration factor for a hemispherical pit is about 2.[4] With change from hemispherical to saucerlike form, the theoretical factor decreases rapidly. For a pitted specimen, the theoretical factor is not the mean of the factors for the individual pits, but is the factor for the pit whose form and size cause the highest effective stress concentration factor, K_F. Although many of the pits are not far from hemispherical, the pit giving the highest theoretical stress concentration factor K probably has less resemblance to a hemisphere than to an oblate spheroid or to a hemiellipsoid of revolution, whose axis of revolution is the minor axis of an ellipse and is in the direction of the principal stress. When the eccentricity ratio for this ellipse is 1.7, which probably is about the maximum for pits formed during the first few days of corrosion, K is about 3.

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[4] A fuller discussion of theoretical and effective stress concentration factors is given in a previous paper (18).

Even in the early stages of corrosion, however, the pits are not entirely smooth. With growth of the pits, projections appear on the boundaries. Although sharp conical projections may cause high values of the theoretical stress concentration factor, the significant value of this factor for stressless corrosion of steels in well water probably does not exceed 3 or 4. Although this is due partly to the fact that, as the projections develop, the pits tend to change from hemispherical to saucerlike form, it is due largely to the fact that the sharp, rootlike projections are generally small.

The stress concentration around a pit may also be influenced by the nearness of another pit. If the line of centers of two pits (near together) is in the direction of the principal stress, each pit has a shielding effect on the other, and the stress concentration is less than if the pits were far apart. If the line of centers is in the transverse direction, however, the stress concentration is greater than if the pits were far apart. (Such a condition tends to cause merging of adjacent pits, when corrosion and cyclic stress are simultaneous.)

The Influence of the Size of a Notch on the Effective Stress Concentration Factor:

The discrepancy between K and K_F tends to increase with the theoretical stress concentration factor and to decrease with increase in the absolute size of the notch. This relation is due to the influence of K and of the size of the notch on the steepness of the theoretical stress gradient. The steepness of the theoretical stress gradient increases with increase in K and with decrease in the size of the notch. A suitable index of steepness of the gradient, suggested by the authors in a previous paper (18), is $(K - 1)/r$, in which r represents the root-radius of the notch.

The available evidence indicates that the discrepancy between K and K_F tends to increase with the steepness of the stress gradient. The fracture of a metal, either by single or repeated loading, apparently is not determined by the highest stress in the theoretical gradient (at a geometrical line), but by the mean stress over an appreciable width of the gradient. The steeper the gradient, therefore, the greater is the difference between this mean stress and the highest stress of the theoretical gradient, and the greater is the difference between K and K_F. The gradient may even be so steep and narrow that the stress peak has practically no effect on the fatigue limit. (Space is not available for discussing the reasons for this behavior of metals.)

Although a sharp, rootlike projection on a corrosion pit may cause a very high value of K, this value is associated with a very low value of r. Such a projection, therefore, gives a very high value of $(K - 1)/r$, the index of steepness of the stress gradient. The sharp projection superposes a very steep, narrow gradient on the broader, less steep gradient induced by the general form of the pit. The stress peaks at such projections may be so sharp that they have no important effect on the fatigue limit. Consequently, when a projection is very small in relation to the size of a corrosion pit, the only significant value of the theoretical stress concentration factor is that based on the general form of the pit.

Correlation Between the Effective Stress Concentration Factor and the Pits Caused by Stressless Corrosion:

From the curves in Fig. 1 have been derived other curves representing the variation of the effective stress concentration factor with the duration of stressless corrosion. These curves (Figs. 2 and 3) are qualitatively the inverse of the curves of Fig. 1. Unlike the curves of Fig. 1, however, the curves of Figs. 2 and 3 all start from the same origin, representing a value of 1.0 for K_F. The differences in course of the curves are due to differences in one or more of at least three factors. These factors are: the form and distribution of the corrosion pits, the size of the pits, and the notch sensitivity of the steel. The notch sensitivity is a function of the chemical composition, mechanical treatment, and heat treatment. In general, the notch sensitivity increases with the tensile strength, although steels of the same tensile strength but of different compositions may differ in notch sensitivity. In correlating the forms and sizes of corrosion pits with the effective stress concentration factor, therefore, allowance must be made for differences in notch sensitivity.

In Fig. 2 are assembled curves representing three steels differing little in tensile strength. They would, therefore, be expected to differ little in notch sensitivity, although the notch sensitivity of the two nickel steels may possibly be a little greater than that of the carbon steel. The divergence of the four curves representing the carbon steel and the two curves representing nickel steels, consequently, can be attributed only to differences in form, size, and distribution of the corrosion pits. For convenient correlation with the corrosion pits in Figs. 4 to 15, symbols have been used in Fig. 2 to indicate specimens that have been examined for corrosion pitting.

Two of the curves in Fig. 2 represent results of corrosion in a stream of well water. One of these curves was obtained with the carbon steel JR-W-10 and the other was obtained with nickel steel IW-W-10. The curve obtained with the nickel steel is much higher in the diagram than the curve obtained with the carbon steel. In a previous paper (**17**), in the absence of information about the corrosion pits, a corresponding difference between curves for nickel and carbon steels was surmised to be due largely to a difference in notch sensitivity. This surmise was made because of statements in the literature that stressless corrosion pitting in steels is little affected by small percentages of alloying elements. As will now be shown, however, these views must be modified.

As shown in Figs. 6 and 7, the differences in chemical composition have had no perceptible influence on the rate of increase of the size of the pits. The change from hemispherical to saucerlike form, however, evidently was much slower for nickel steel IW-W-10 than for carbon steel JR-W-10. Hemispherical pits are found in the nickel steel after corrosion for 100 days (Fig. 7 (*b*)), and deep saucerlike pits are found after corrosion for longer times (Fig. 7 (*c*) and (*f*)). Specimens of carbon steel JR-W-10, however, show only saucerlike pits after corrosion for 59, 69, and 93 days (Fig. 6 (*g*) and (*h*) and Fig. 7 (*a*)). The difference in rapidity of the change from hemispherical to saucerlike form of the pits in the nickel steel and carbon steel accounts for the difference in rapidity of the approach of the corresponding curves to a horizontal direction in Fig. 2.

In Fig. 3, the curves representing corrosion of carbon steel and nickel steel in well water are repeated (as broken curves) for comparison with curves representing corrosion of four other steels in well water. Views of pits in one of these steels (chromium-nickel steel AX-W-10) are shown in Figs. 6 and 7. After corrosion for 2, 4, and 47 days (Fig. 6 (*a*), (*b*), and (*e*)) the specimens show hemispherical pits. After corrosion for 84, 150, and 220 days (Fig. 6 (*i*) and Fig. 7 (*d*) and (*e*)), the pits found are of saucerlike form. This change of the pits from hemispherical to saucerlike form evidently is somewhat more rapid in chromium-nickel steel AX-W-10 than in nickel steel IW-W-10, but is less rapid than in carbon steel JR-W-10 (Figs. 6 and 7). To this difference in the rate of change from the hemispherical to the saucerlike form may be attributed the fact that the curve representing steel AX-W-10 is below the curve representing steel IW-W-10, in spite of the greater tensile strength of steel AX-W-10.

Another curve in Fig. 3 was obtained by corrosion of nickel steel IW-14.5 in well water. As this steel differed only in heat treatment from steel IW-W-10, the corrosion pits in steel IW-14.5 (not examined) probably were about the same as those found after corrosion of steel IW-W-10 for corresponding times. The great difference between the curves for steel IW-W-10 and IW-14.5, therefore, must be attributed to a difference in notch sensitivity, due to the difference in hardness of the two steels. A difference in notch sensitivity probably accounts also for the difference between the curves for the two carbon steels, JR-W-10 and AT-W-10.

The highest curve in Fig. 3 represents the steel of highest tensile strength. As chromium-nickel steel AY-W-10 is only slightly stronger than chromium-nickel steel AX-W-10, however, these two steels probably differ little in notch sensitivity. The great difference in position of the two curves in Fig. 3, therefore, probably is due in part to a difference in the corrosion pitting (not examined).

As the highest and lowest curves in Fig. 2 were obtained with steels of about the same composition and differing little in tensile strength, the great difference between these curves cannot be due to a difference in the notch sensitivity. As already shown, the pits tend to grow more slowly and to spread relatively more in distilled water than in well water. The great difference between the corresponding curves in Fig. 2, therefore, must be attributed chiefly to the influence of size of the pits on the effective stress-concentration factor.

The divergence of the four curves representing carbon steel JR-W-10 (Fig. 2) can be attributed only to differences in pitting under the four different corrosion conditions. The two most widely diverging curves are those representing corrosion in streams of well water and Severn River water. Comparison of Fig. 6 (*g*) and (*h*) and Fig. 7 (*a*) with the views in Fig. 11 shows that the pits tend to be broader but not much deeper after corrosion in Severn River water than after corrosion for corresponding times in well water. The theoretical stress concentration factor, based on the general form of the corrosion pits and without regard for their distribution, therefore, would be greater for the pits caused by corrosion of this carbon steel in well water than for the pits caused by corrosion in Severn River water. Although sharp rootlike projections from pits caused by corrosion in Severn River water would tend to increase the theoretical stress concentration factor, they probably were too small to have much influence on the effective stress concentration factor. The greater effective stress concentration caused by corrosion in Severn River water (Fig. 2) may be due largely to the greater size of the corrosion pits, especially of a few of the largest pits (compare Fig. 10 with Figs. 4 and 5). The effect of size probably more than compensated for the difference in theoretical stress concentration (due to separate pits). In the narrow regions between the broad pits caused by corrosion in Severn River water, moreover, the theoretical stress concentration may have been high. The relatively high position of the corresponding curve in Fig. 2, therefore, probably is due to the relatively large size of the pits and to high theoretical stress concentration in the narrow regions between such pits.

Throughout the first 60 days, the effective stress concentration due to corrosion in a spray of normal salt solution was only slightly greater than that due to corrosion in a stream of well water. This relation was found for carbon steels JR-W-10 and AT-W-10 and for chromium-nickel steel AX-W-10. The roughly hemispherical pits caused by corrosion in the salt spray (Fig. 15) were very small in comparison with the pits caused by corrosion in a stream of well water (Figs. 6 and 7). If the effective stress concentration were due to these hemispherical pits, therefore, it would be greater after corrosion in well water than after corrosion in the salt spray. Such a relation was actually found after corrosion for 48 days in salt spray (Figs. 1 (*c*) and 2). At this time, however, there were no rootlike projections. Soon after the appearance of these projections in the carbon steel specimens, the effective stress concentration began to increase at an increasing rate (see curve for JR-W-10 in Fig. 2). The theoretical stress concentration factor caused by a rootlike projection alone probably would differ little from that caused by a narrow cylindrical cavity and would be about 3. Such a projection from a much larger hemispherical pit would cause a value of about 6 for K. Because of the small size, however, the effective stress-concentration factor K_F, was much less, but was increasing rapidly with the development of the projections. In the chromium-nickel steel AX-W-10, no rootlike projections appeared (Fig. 15 (*f*)), and the curve of effective stress concentration showed no reversal (Fig. 3).

After corrosion in a spray of softened water, the effective stress concentration factor was much greater than after corrosion in a spray of normal salt solution. This relation, which was found with three steels (Figs. 2 and 3), may be attributed to the fact that the hemispherical and saucerlike pits were much larger after corrosion in the softened water (Fig. 13) than after corrosion in the salt spray (Fig. 15). However, the growth of the rootlike projections, due to corrosion of the carbon steels in the salt spray, might eventually cause the corresponding curves to cross the curves representing corrosion in the spray of softened water (Figs. 2 and 3).

The pits caused by corrosion in the spray of softened water (Fig. 13) resemble the pits caused in the same steels by corrosion in a stream of well water (Fig. 6 (*i*) and Fig. 7 (*d*) and (*e*)). The effective stress concentration caused by corrosion in a spray of softened water, however, was greater than that caused by corrosion in a stream of well water, and was nearly as great as that caused by corrosion in a stream of Severn River water. This relation probably is due to the relatively large size of some of the pits caused by corrosion in a spray of softened water (compare Figs. 5 and 12).

For steels differing little in tensile strength, the differences in the effective stress concentration factor due to corrosion under the different conditions, exclusive of corrosion in a stream of distilled water, are not great. During the early stages of corrosion, these differences in K_F are easily accounted for by the observed differences in the form and size of the individual pits. The difficulties of correlation naturally increase as the pitting spreads over the surface. The comparison of Figs. 2 and 3 with the views of corrosion pits (Figs. 4 to 15), however, confirms the conclusion that the effective stress concentration factor is a function of the form, size, and distribution of the corrosion pits and of the notch sensitivity of the steel.

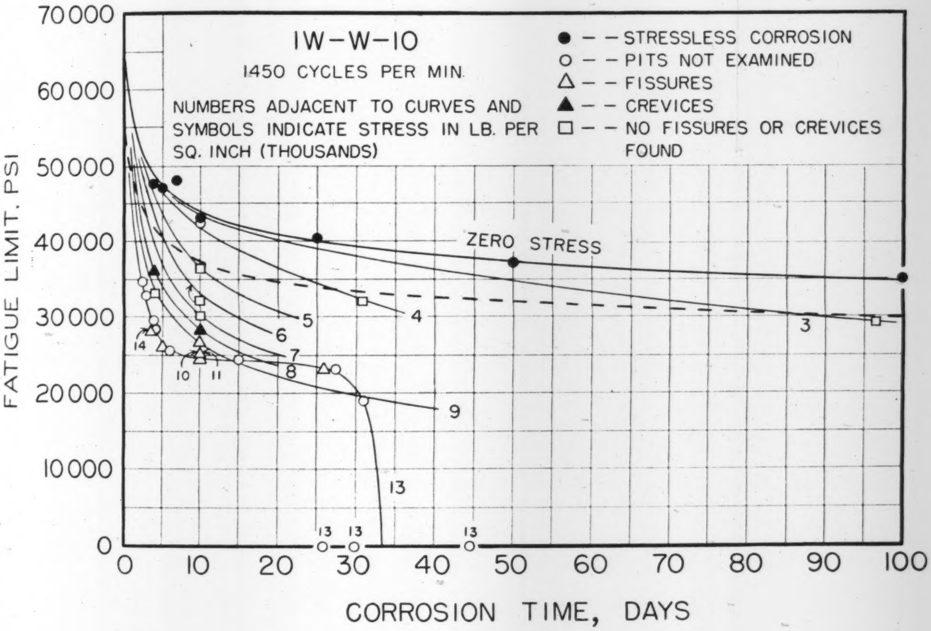

FIG. 16.—The Lowering of the Fatigue Limit of a Nickel Steel Due to Stress Corrosion in Well Water.

V. PITS CAUSED BY STRESS CORROSION OF STEEL IN WELL WATER

Figures 17 and 18 show surface and sectional views of specimens of nickel steel IW-W-10 after corrosion with 1450 stress cycles per minute under various cyclic stresses and for various times. Comparison of the views in each figure, therefore, reveals the influence of both cyclic stress and corrosion, with constant cycle frequency. The views in each figure are arranged primarily in the order of decreasing cyclic stress and secondarily in the order of increasing corrosion time.

In the surface views (Fig. 17), some pits appear round, like the pits caused by stressless corrosion (Fig. 4). Other pits consist of round or irregular cavities apparently crossed by cracks, which are transverse to the direction of principal tensile stress. The corresponding sectional views at higher magnification (Fig. 18), however, show that these are not cracks but are sharp, deep corrosion pits; they are filled with corrosion products similar to those found in round pits. The sharp, deep pits are found at the high-stress end of the series of views; at the low stress end of the series, the pits are entirely of the rounded form. The magnitude of the stress, therefore, is one of the factors determining whether the sharp projections shall be formed. Another factor, as will be shown, is the corrosion time.

The first stages of the development of projections of this form cannot be determined by examination of surface views, especially at low magnification. Even when such projections do not appear on the surface, an examination of a longitudinal section may reveal sharp, wedge-shaped projections extending inward

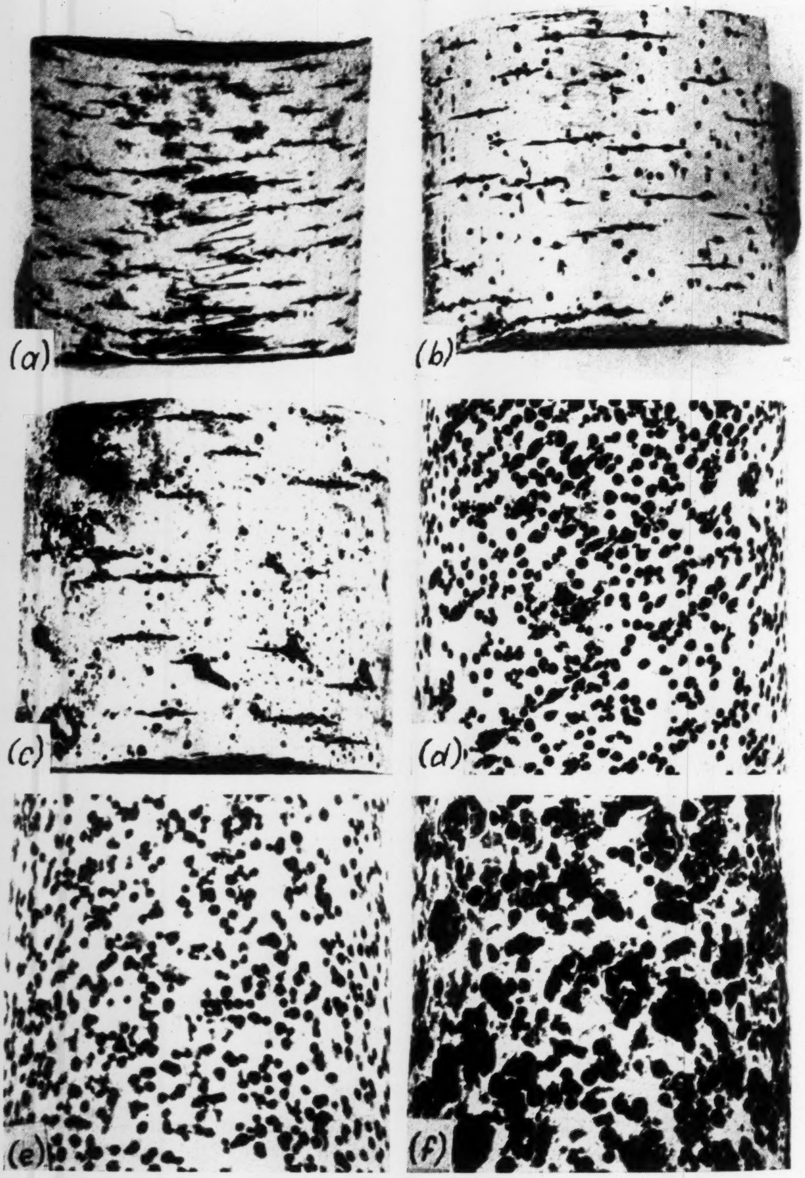

Fig. 17.—Nickel Steel IW-W-10, 1450 cycles per minute, Well Water, Outer Surfaces (× 4).

(a) 13,000 psi., 5 days
(b) 10,000 psi., 10 days
(c) 9000 psi., 10 days
(d) 8000 psi., 10 days
(e) 4000 psi., 31 days
(f) 3000 psi., 97 days

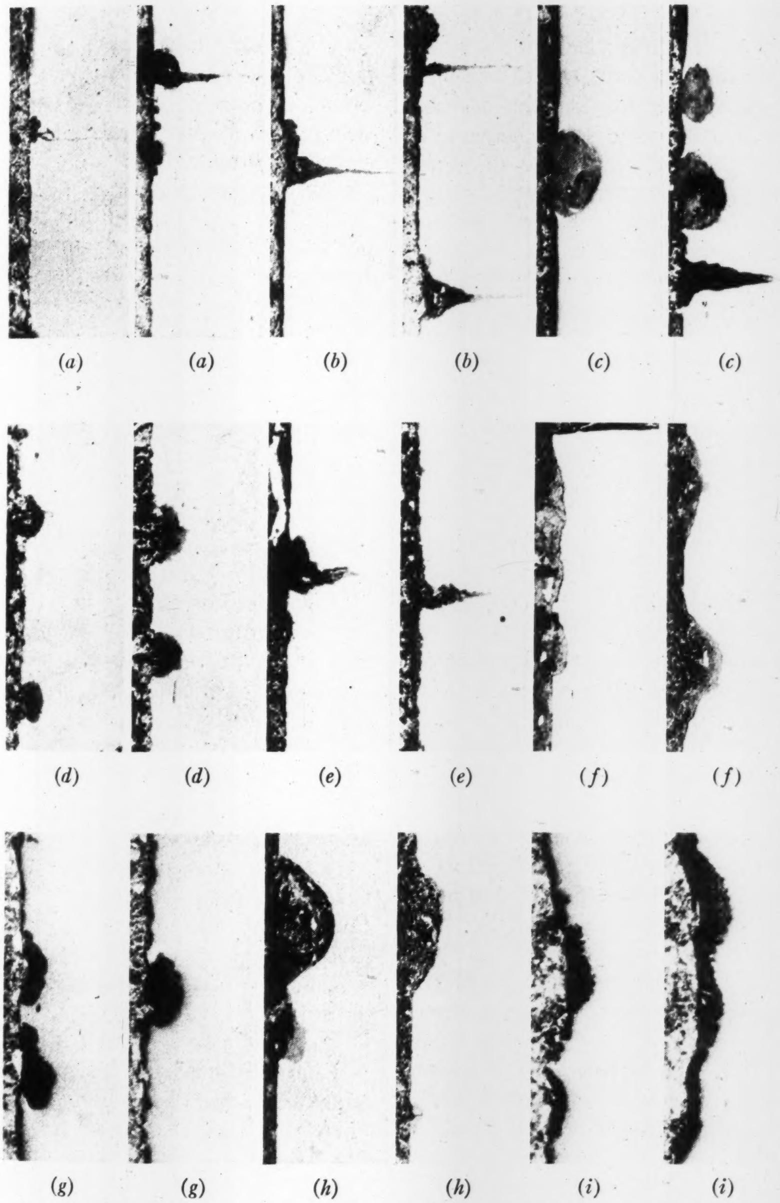

FIG. 18.—Nickel Steel IW-W-10, 1450 cycles per minute, Well Water, Longitudinal Sections (× 50).

(a) 14,000 psi., 3.2 days
(b) 13,000 psi., 5 days
(c) 10,000 psi., 10 days
(d) 9000 psi., 4 days
(e) 9000 psi., 10 days
(f) 8000 psi., 10 days
(g) 7000 psi., 10 days
(h) 4000 psi., 31 days
(i) 3000 psi., 97 days

from rounded pits. The sharp, transversely extended pits visible in surface have been called fissures (**18**); the wedge-shaped pits not visible on the surface have been called crevices. A crevice sometimes is merely an early stage in the development of a fissure.

The influence of stress on the development of fissures is clearly revealed by comparison of Fig. 17 (*b*), (*c*), and (*d*), and Fig. 18 (*c*), (*e*), (*f*), and (*g*). These views show the effects of various cyclic stresses acting during the same corrosion time, 10 days. For stresses of 9000 psi. or more, round or irregularly extended pits are traversed by fissures. In the views representing stresses of 8000 and 7000 psi., the pits appear round, like the pits caused by stressless corrosion. Fissures evidently do not appear during 10 days corrosion at 1450 cycles per minute when the cyclic stress is less than about 9000 psi. At stresses of 4000 and 3000 psi. (Fig. 17 (*e*) and (*f*) and Fig. 18 (*h*) and (*i*)), fissures do not appear during corrosion for much longer times. At sufficiently high stress (Fig. 17 (*a*) and Fig. 18 (*a*) and (*b*)), fissures may appear after corrosion for much less than 10 days.

The absence of rounded pits from some of the sections of fissures (Fig. 18) does not mean that the fissures started inward directly from the surface. All the fissures, as shown in the surface views (Fig. 17), have started at rounded corrosion pits. From these origins, the fissures have extended both transversely and inward. Some of the sections in Fig. 18 are nearly at the centers, and some are at the edges of the rounded origins of the fissures. Other sections are at a distance from these origins.

The evidence, therefore, indicates that stress corrosion pits, in steel corroded in well water, are modifications of pits caused by stressless corrosion. The number of pits per unit area, and their distribution, are determined by the same uncontrollable variables that determine the number of pits formed by stressless corrosion. Great differences, therefore, may be found in the number and distribution of these anodic regions after apparently identical corrosion conditions. The size of the pits, moreover, generally varies inversely with the number.

The fissured pits (Fig. 17) generally are much larger than pits formed in corresponding times by stressless corrosion. Cyclic stress evidently tends to increase the size of the corrosion pits. The accelerating effect of cyclic stress on corrosion, moreover, evidently tends to be greatest at the equator[5] of a roughly hemispherical corrosion pit. The tendency to form sharp equatorial fissures may be attributed to the fact that nearly all the concentration of stress is in a narrow equatorial ring (**18**). With development of a fissure, the stress concentration is intensified.

To facilitate comparison between the corrosion pits shown in Figs. 17 and 18 and the corresponding net damage, the forms of the corrosion pits are classified by means of the symbols used to designate the experimental points in Fig. 16. Different symbols are used to designate fissures, crevices, and rounded pits. The fissures caused by corrosion for 10 days at stresses of 10,000 and 9000 psi. (Fig. 17 (*b*) and (*c*) and Fig. 18 (*c*) and (*e*)) have lowered the fatigue limit from 64,000 psi. to 25,000 and 26,000 psi. (Fig. 16). These values are far below the fatigue limit (44,000 psi.) that would be found after 10-day corrosion without stress. The net damage caused by crevices tends to be intermediate between the damage caused by fissures and that caused by rounded pits. Considerable net damage may be caused by stress corrosion, even if no crevices or fissures have appeared. This is illustrated by the positions of the points representing the effects of corrosion for 10 days at stresses less than 8000 psi.

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[5] The polar axis is assumed to be in the direction of the principal tensile or compressive stress.

Pitting Caused by Stress Corrosion of Steel in Distilled Water

Figure 20 shows surface views of specimens of nickel steel LQ-W-10 after corrosion in distilled water under various stresses, with frequency of 1450 cycles per minute. All but one of the specimens that are shown in these surface views are shown in sectional views in Figs. 21 and 22.

In Fig. 20 (*a*), very short fissures are visible, starting from some of the rounded pits. Even very small round pits, such as those in the cluster near the lower right corner of this view, are origins of fissures, clearly visible at higher magnification. The early start of these fissures may be attributed to the high cyclic stress (40,000 psi.). After somewhat longer time at lower stress (Fig. 20 (*b*)), the fissures are further advanced. No fissures are visible in Fig. 20 (*c*) and (*d*), although Fig. 17 (*a*) and (*b*) shows that prominent fissures would have developed during corrosion at the same stresses and for the same times in well water. Corrosion for much longer times in distilled water at much lower stresses, however, has caused prominent fissures (Fig. 20 (*e*) and (*f*).

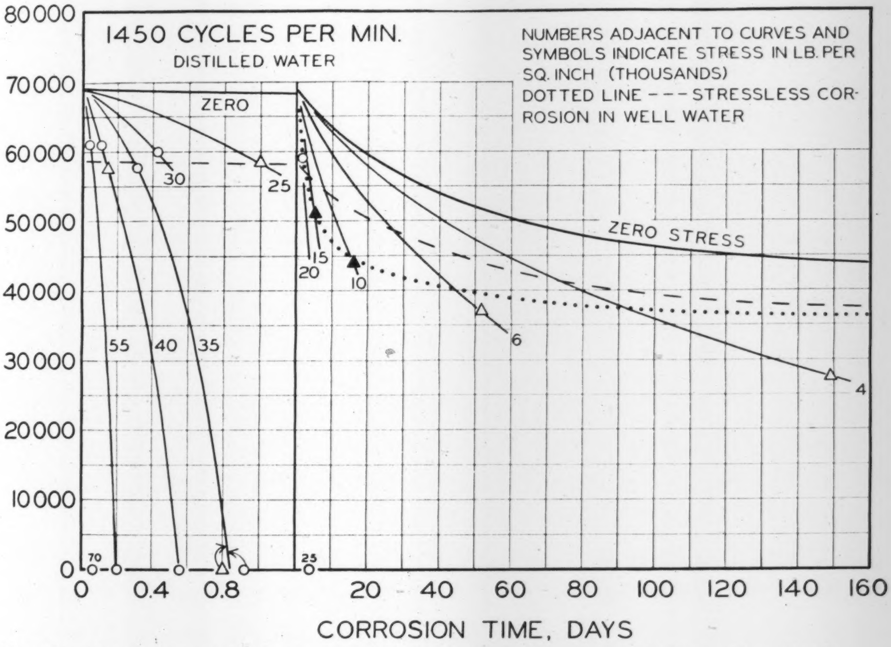

Fig. 19.—The Lowering of the Fatigue Limit of a Nickel Steel Due to Stress Corrosion in Distilled Water.

Although no fissures are visible in Fig. 20 (*c*) and (*d*), crevices are visible in one of the corresponding sectional views, Fig. 21 (*c*). Fissures visible in Fig. 20 (*b*), (*e*), and (*f*) are also revealed by sectional views in Fig. 21 (*a*) and (*d*) and Fig. 22. The evidence indicates that a higher stress or a longer time is required for the development of fissures by stress corrosion in distilled water than by stress corrosion in well water.

The sharp transverse fissures caused by corrosion at 40,000 psi. for 0.15 day (Fig. 20 (*a*)) have lowered the fatigue limit from 69,000 to 57,500 psi. (Fig. 19). Although the theoretical stress concentration was large, the effective stress concentration was small because of the small size of the fissures. The larger

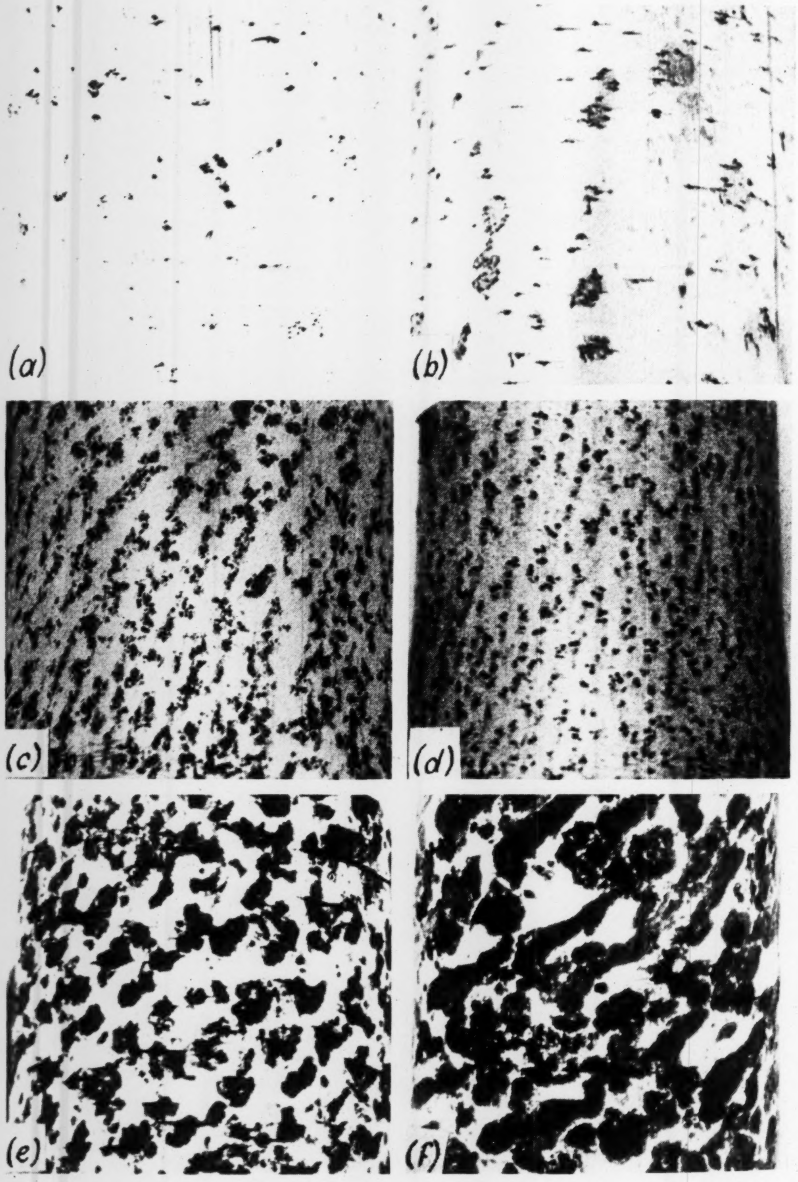

FIG. 20.—Nickel Steel LQ-W-10, 1450 cycles per minute, Distilled Water, Outer Surfaces (× 4).

(*a*) 40,000 psi., 0.15 day
(*b*) 35,000 psi., 0.8 day
(*c*) 15,000 psi., 5.1 days
(*d*) 10,000 psi., 16 days
(*e*) 6000 psi., 52 days
(*f*) 4000 psi., 149 days

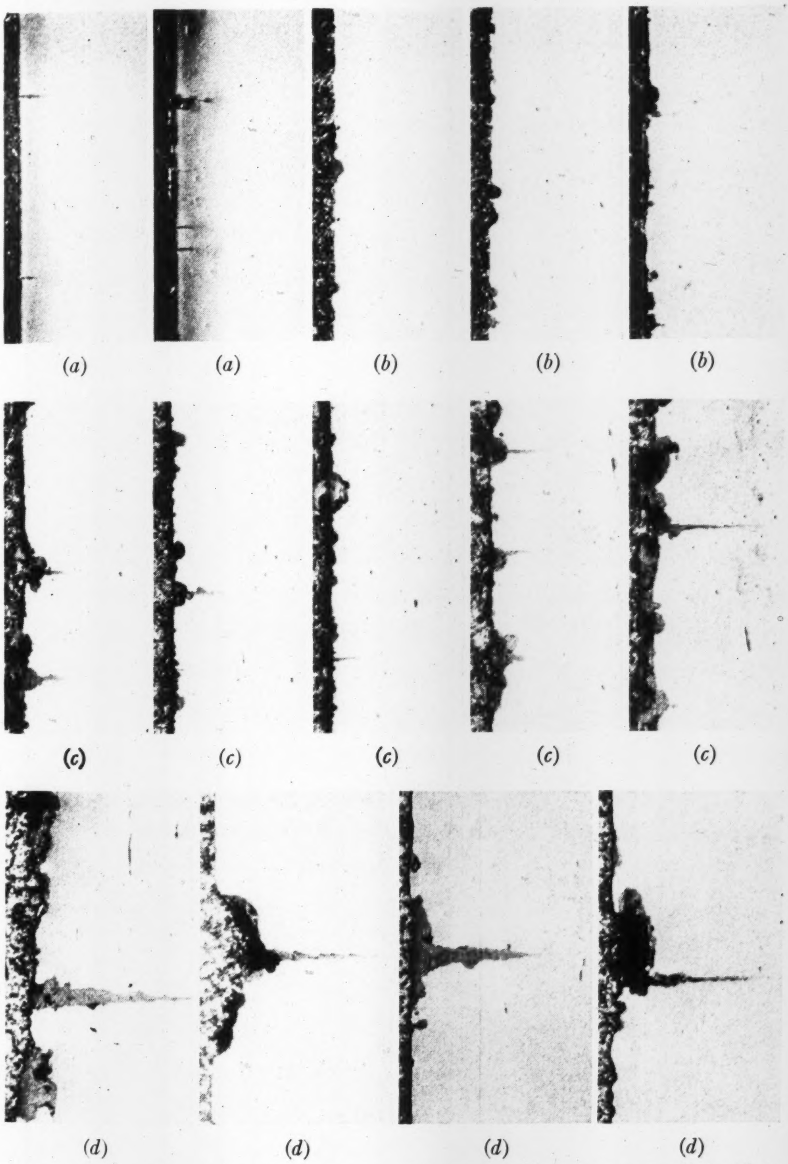

FIG. 21.—Nickel Steel LQ-W-10, 1450 cycles per minute, Distilled Water, Longitudinal Sections (× 50).

(a) 35,000 psi., 0.8 day
(b) 15,000 psi., 5.1 days
(c) 10,000 psi., 16 days
(d) 6000 psi., 52 days

fissures due to corrosion at 35,000 psi. for 0.8 day evidently caused much greater effective stress concentration (Fig. 20 (b) and Fig. 21 (a)). As shown in Fig. 19, this specimen failed by fatigue during corrosion. Much total and net damage also was caused by crevices such as those shown in Fig. 21 (c), and by fissures such as those shown in Fig. 20 (e) and (f), Fig. 21 (d), and Fig. 22

(Fig. 19). Considerable net damage, moreover, was caused by the rounded pits and incipient crevices shown in Fig. 21 (*b*).

The evidence in Figs. 19 to 22, therefore, confirms the evidence in Figs. 16, 17, and 18, that cyclic stress tends to accelerate corrosion pitting, especially in regions of highest stress concentration around the corrosion pits. A discus-

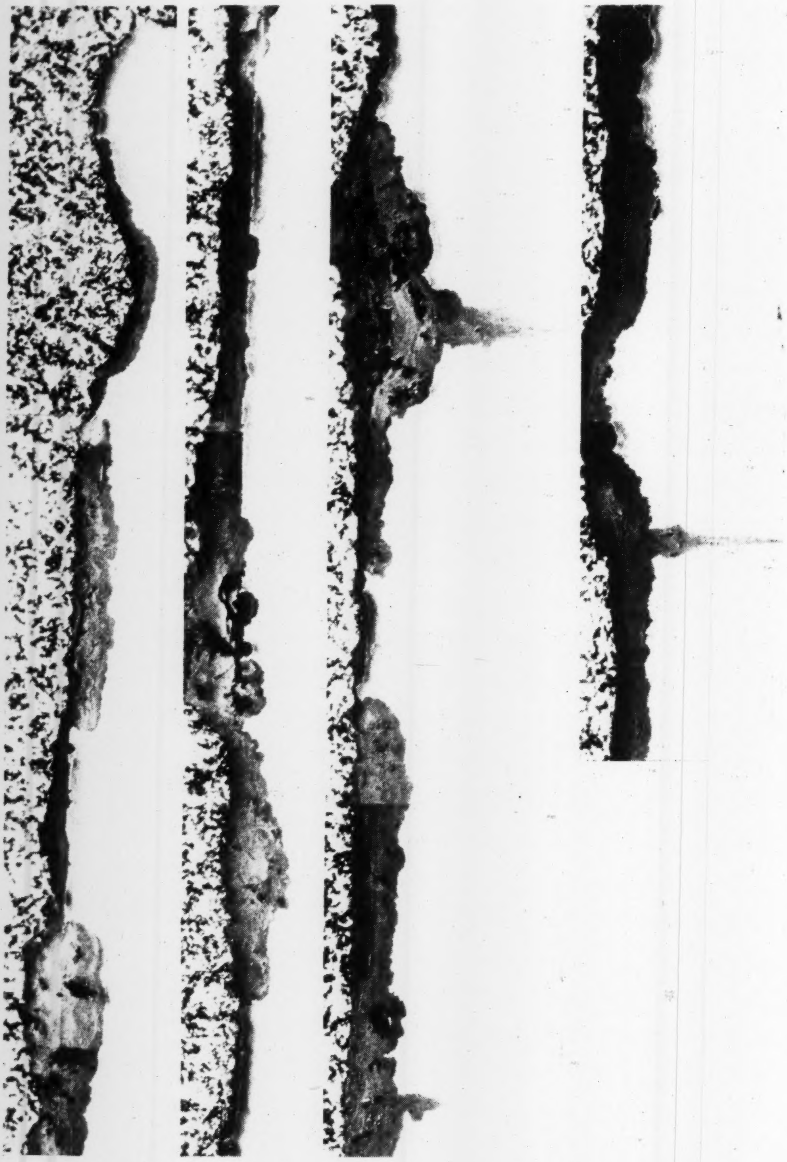

Fig. 22.—Nickel Steel LQ-W-10, 1450 cycles per minute, Distilled Water, Continuous Longitudinal Section (× 50).
4000 psi., 149 days

sion of the process of stress corrosion is given in a previous paper (18).

Acknowledgment:

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(14) D. J. McAdam, Jr., "Influence of Stress on Corrosion," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Division, Vol. 99, p. 282 (1932).

(15) D. J. McAdam, Jr., "Stress Corrosion of Metals," *Proceedings*, Zurich Congress, Internat. Assn. Testing Materials, Vol. 1, p. 228 (1931).

(16) D. J. McAdam, Jr., "Influence of Water Composition on Stress Corrosion," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 259 (1931).

(17) D. J. McAdam, Jr., and R. W. Clyne, "Influence of Chemically and Mechanically Formed Notches on Fatigue of Metals," *Journal of Research*, Nat. Bureau Standards, Vol. 13, p. 527 (1934).

(18) D. J. McAdam, Jr., and G. W. Geil, "Influence of Cyclic Stress on Corrosion Pitting of Steels in Fresh Water, and Influence of Stress Corrosion on Fatigue Limit," *Journal of Research*, Nat. Bureau Standards, Vol. 24, p. 685 (1940).

(19) D. J. McAdam, Jr., and G. W. Geil, "Influence of Stress on the Corrosion Pitting of Aluminum Bronze and Monel Metal in Water," *Journal of Research*, Nat. Bureau Standards, Vol. 26, p. 135 (1941).

DISCUSSION

Mr. J. M. Lessells.[1]—The paper is another contribution to the subject of corrosion-fatigue with which work the name of McAdam will always be associated.

There are indications that the method of machining the original surface may have a bearing on the results obtained. This appears to be true for fretting corrosion. It would, therefore, be of value to know how the test bars were machined and the grade of lubricant employed in the final grinding. The steps in the final polishing would also be of interest.

On page 700, it appears that only one specimen was employed to determine the fatigue life. This is satisfactory provided we know there is normally little "scatter" in the results. Three specimens might have been better than one.

On page 719, reference is made to fracture by a single or repeated load. "Single" presumably refers to brittle material. On this page the author also discusses stress gradient due to concentration effects, but does not enlarge on this due to lack of space. Although this may be fully covered by previous papers by the author some further enlargement of this discussion might be very useful in the present paper.

A final point is that a few plots with a less number of variables might be helpful. For instance from Fig. 3 for a period of 70 days corrosion time in well water we get for the steels designated AT, JR, IW and AY effective stress concentration factors K_F of 1.27, 1.5, 1.78 and 2.0., respectively.

Mr. D. J. McAdam, Jr.[2]—The question of surface preparation of these specimens was very thoroughly described in connection with previous papers on stress corrosion. I would refer Mr. Lessells to these previous papers.

The question of the use of one specimen in determining a fatigue limit might be answered by saying that we at first used a number of specimens to determine the fatigue limit. Later we found that by selecting a stress that would cause failure at not much less than a million cycles, we could determine the fatigue limit by extrapolation. By using only one specimen for a determination we were able to make tests at a number of different stresses, and this was quite an advantage.

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[2] Metallurgist, National Bureau of Standards, Washington, D. C.

A NEW HIGH-TEMPERATURE FATIGUE MACHINE

By W. P. Welch[1] and W. A. Wilson[2]

Synopsis

A new machine for the rapid determination of the endurance limit of metals at high temperatures is described. The specimen is stressed in alternating bending at a frequency of 120 cycles per second (7200 cycles per minute) and is driven at constant amplitude by a reciprocating electromagnetic motor supplied with 60-cycle power from the house line. Since the machine operates very close to resonance, an electronic control is provided to maintain constancy of amplitude despite the small changes in the line frequency (± $\frac{1}{3}$ per cent) which occur on a well-regulated power system. Heating of the specimen is accomplished by means of a simple bell-type electric furnace, and tests may be run at any constant temperature up to 1000 F.

The specimen employed is of generous proportions and is simple in form. No accurate alignment of parts is required in setting up the specimen in the machine.

The results of operating experience over a period of a year is reported along with test results showing the quality of the data that have been obtained.

To the designer of high-speed machinery the endurance limit is one of the most significant physical properties of the materials used for highly stressed parts. Although steels and other materials have for many years been employed in high-temperature applications under conditions of repeated stress, at present very little information is available on the endurance limit of these materials at elevated temperatures. New high-temperature alloys in great abundance are now being offered the designer, yet in almost all instances the endurance limit of these alloys at their proposed operating temperature is estimated from room temperature tests.

There is, of course, a very good reason for this deficiency. The conventional room temperature determination of the endurance limit is in itself a very time-consuming and expensive procedure, involving the testing of from five to twenty specimens, the running time of several of the tests being of the order of weeks. Now if we add another parameter, the temperature, to these experiments, an enormous amount of time is required for the complete test. Still another fact makes the high-temperature test more laborious. In general, and especially for the case of ferrous alloys, the conventional *S-N* curve depicting the results of a complete fatigue test becomes almost straight and horizontal at a number of cycles usually not exceeding 10,000,000 cycles, indicating that an endurance limit has been reached. However, this is not the case for these same materials at high temperatures. Experimental evidence, some of which will be presented herein, shows that the *S-N* curve continues to fall even beyond 100,000,000 cycles. In fact, for some of the materials which we have tested at 850 F. the curve is still falling at 500,000,000 cycles. These two factors, the inclusion of an additional parameter, and the necessity for making very long tests, make high-temperature fatigue testing very lengthy even when compared with fatigue testing at room temperature.

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[2] Mechanical Engineer, Turbine Engineering Dept., Westinghouse Electric and Manufacturing Co., Lester, Pa.

Several elevated temperature fatigue machines have been developed in recent years by other investigators.[3] For the most part, these operate at low or medium speeds, and, inasmuch as we desired to subject specimens to as many as a billion stress cycles (1 yr. at 1900 cycles per minute), these machines were considered too slow for our purpose. The B. P. Haigh electromagnetic fatigue machine[4] may be adapted to high-speed operation and testing at elevated temperatures, but it was desired to test a larger specimen than the Haigh specimen and also to eliminate the alignment difficulties intrinsic with the axial loading type of machine. The major characteristics which are desired in a fatigue machine for this purpose may be summarized as follows:

1. It should be possible to run tests at any constant temperature from room temperature to 1000 F.
2. The speed should be high, preferably greater than 100 cycles per second, but not so great as to affect the endurance limit to a marked extent.
3. The specimen should be simple in form. Complicated clamping schemes should be avoided because of the high relaxation rates encountered at elevated temperatures.
4. Very accurate alignment of the specimen should not be required. In this respect the reversed-bending type of machine would be the most suitable.
5. The machine should preferably be of the constant-load type.

To fulfill this need for high-temperature testing, a new fatigue machine has recently been developed. The specimen is stressed in alternating bending at a frequency of 120 cycles per second (7200 cycles per minute), and, by means of an electric furnace, tests may be conducted at any temperature up to 1000 F. As shown in Fig. 1, the specimen is of generous size and moreover it is easily machined. A comparison of this specimen with two other common types of fatigue specimens, one for the B. P. Haigh direct stress machine and the other for the R. R. Moore rotating beam machine may be obtained from Fig. 2. The complete machine including the control, is shown in Fig. 3.

PRINCIPLES OF OPERATION

The machine may be classed as a fixed-cantilever, constant-deflection, type, designed primarily for the application of alternating bending stress to the specimen.[5] Figure 4 is a diagrammatic sketch showing the moving parts of the machine and illustrating the operating principles. The specimen *A* is clamped at the top to the stationary frame *B* and at the bottom by the lower clamp *C*. This specimen-clamp system is flexed in bending at a constant alternating amplitude by a reciprocating driving motor, connected to *C* by the drive rod *D*. The driving motor comprises the armature beam *E*, shown in elevation in Fig. 4 (*a*) and (*b*), and a stator, whose plan view is Fig. 4 (*c*). The armature beam is constrained to rotation about an axis through *O* by the torsion bar *F*. When power is fed into the stator coils alternating magnetic forces, indicated by the double arrows in Fig. 4 (*a*), act on the armature punchings *G*. These forces drive the system at the desired double amplitude *d*.

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[3] H. F. Moore and N. J. Alleman, "Progress Report on Fatigue Tests of Low-Carbon Steel at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 114 (1931).

F. M. Howell and E. S. Howarth, "A Fatigue Machine for Testing Metals at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 206 (1937).

[4] For a description of this machine see "The Haigh Alternating Stress Testing Machine," *The Engineer*, Vol. 132, July 29, 1921, pp. 116-117.

[5] Means are provided for the superposition of a steady bending stress to the specimen, but as yet no operating experience has been acquired with this feature.

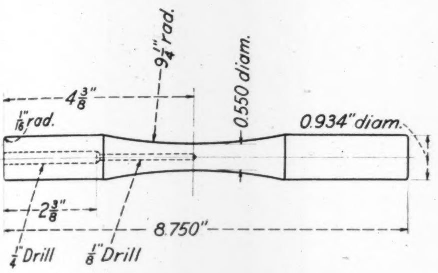

FIG. 1.—Specimen for 120 cycle Bending Fatigue Machine.

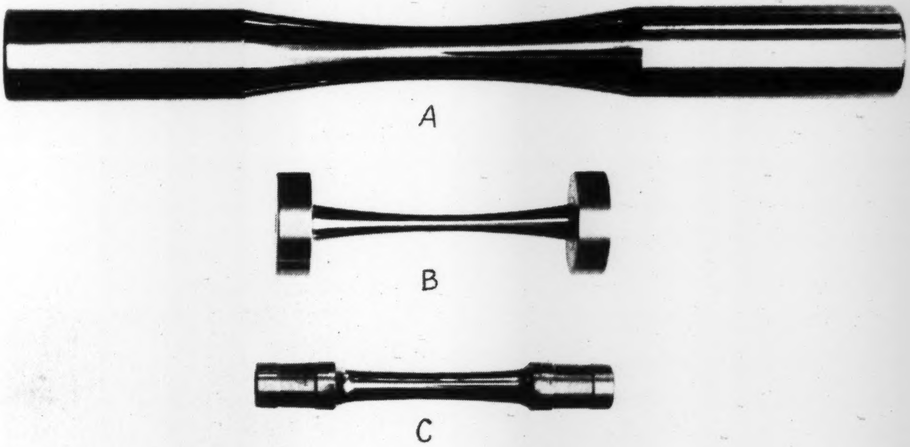

FIG. 2.—Comparison of Specimen (*A*) with Specimens for B. P. Haigh Machine (*B*) and R. R. Moore Machine (*C*).

Two-phase 60 cycles operating power is obtained through Scott transformers from the 60-cycle house line. The electromagnetic forces, since they depend only upon the magnitude and not the direction of the current, vary at twice the line frequency, or 120 cycles per second. To reduce the magnitude of the required driving forces, the armature-specimen system is tuned to nearly 120 cycles by the addition of small tuning weights to the lower clamp at *H*. Furthermore, to reduce the force transmitted by the drive rod *D*, the vibrating system consisting of the armature beam *E* and the torsion spring *F* is tuned alone to 120 cycles. Thus, the machine operates very close to resonance, and, as a result, small changes in the line frequency will cause large changes in the double amplitude *d*, provided, of course, that no control is employed. Actually, these and other changes in amplitude are inhibited by means of an electronic control circuit whose input connects across the two contacts, *L* and *L'* Fig. 4 (*a*). Contact *L* is mounted on the armature beam, while contact *L'* is adjustably mounted onto the frame. The control, described in Appendix A,[6] regulates the driving power, which is normally under 40 watts, so that the contacts strike lightly when the beam

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[6] See p. 743.

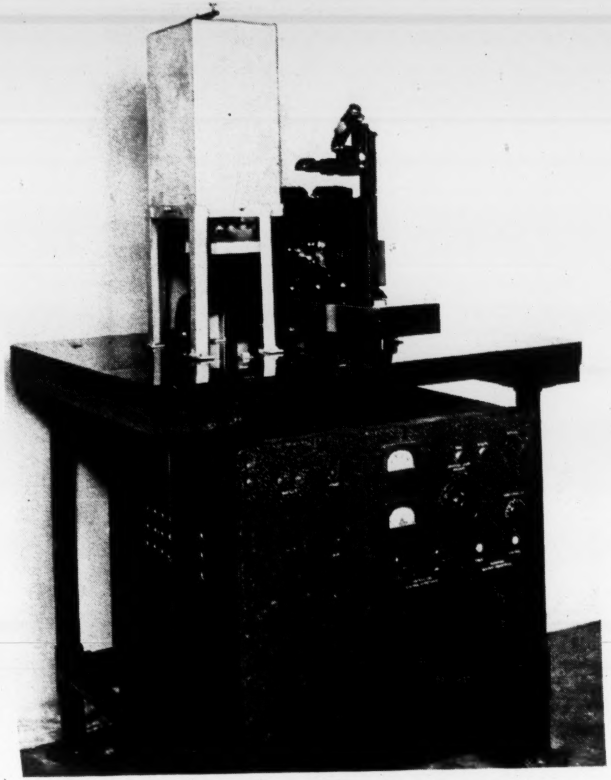


FIG. 3.—Front View of Complete Machine.

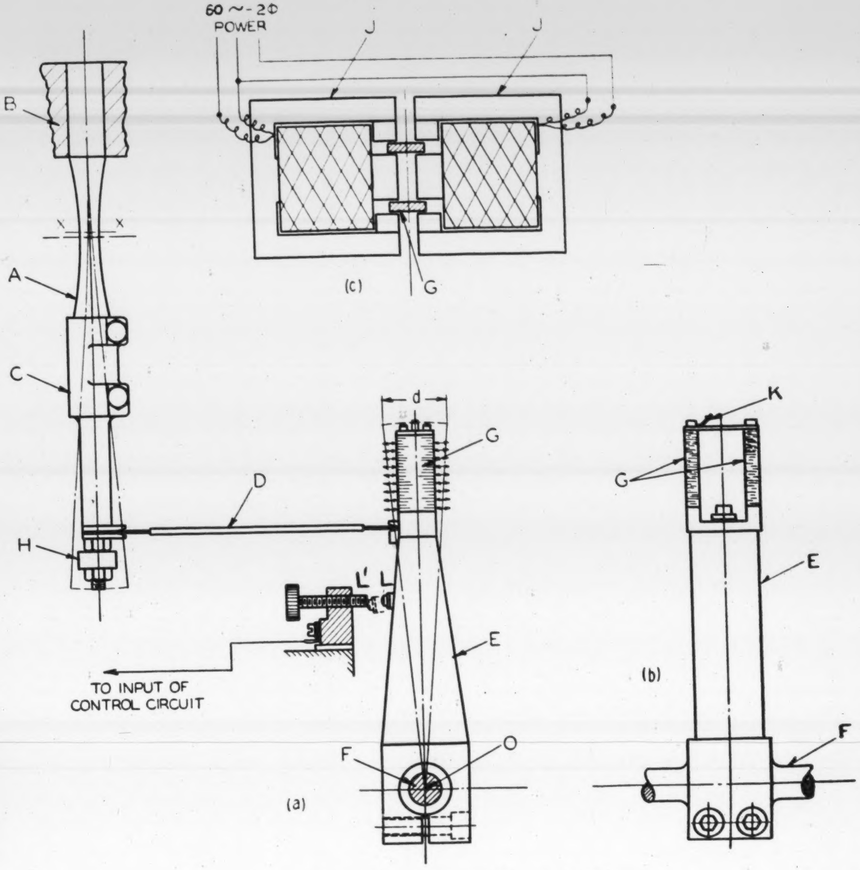

60 ~-2Φ
POWER
J
J
(c)
G
B
x
x
A
C
H
D
d
G
K
G
L'
L
E
E
TO INPUT OF
CONTROL CIRCUIT
(b)
F
F
O
(a)

FIG. 4.—Schematic Diagram of Mechanical Parts.

is in the extreme left-hand position. The double amplitude *d* may be varied by changing the position of the contact *L′* by means of the adjusting screw.

CONSTRUCTION DETAILS

Figure 5 is a good close-up view of the mechanical parts and the driving motor. Shown in the foreground is the calibrated microscope used for reading the double amplitude of motion *d*. Mounted on the top of the armature beam is a target consisting of a small gold bead attached to an inclined flat plate, *K*, Fig. 4 (*b*). This target, when suitably illuminated, appears in the microscope as a bright line, the length of which is the double amplitude *d*, and this length may be easily read from a calibrated scale in the microscope eyepiece. The crank protruding from the middle of the machine regulates the position of the adjustable contact which controls the operating amplitude.

A better idea of the constructional features may be obtained from the assembly drawings, Fig. 6. The electric furnace 4 is a bell-type furnace which slips over the frame 2 and rests on the furnace supporting stand 5. The furnace is maintained at a constant temperature by means of a differential expansion relay 6 consisting of a 18-8 stainless steel tube surrounding a quartz rod. The differential expansion between tube and rod opens and closes a pair

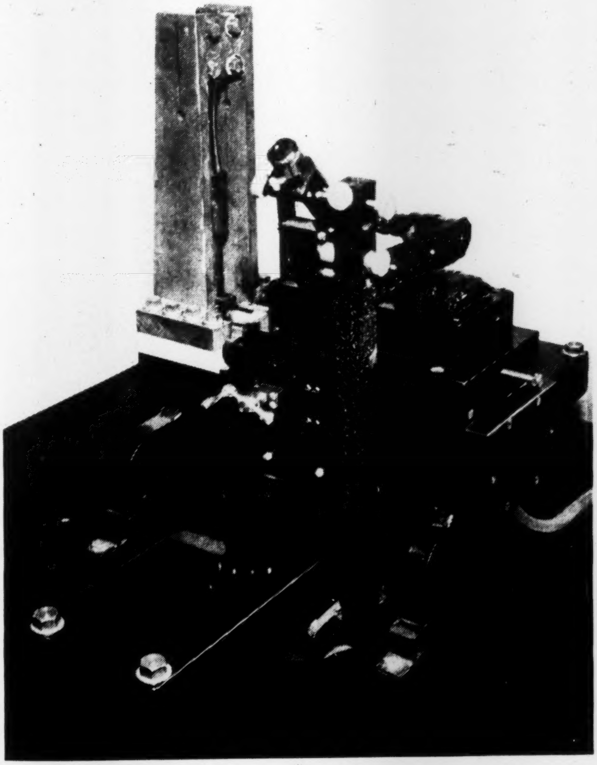
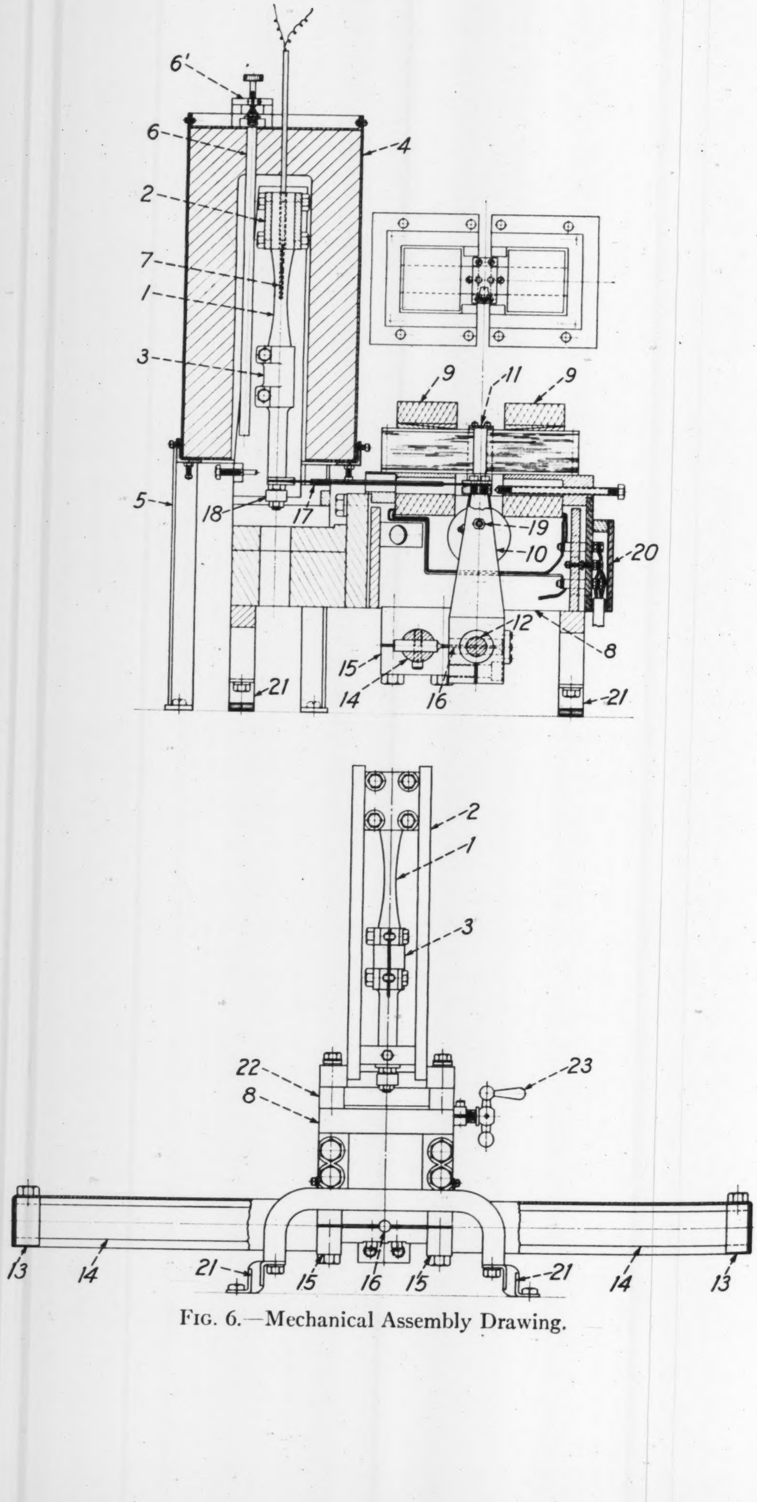


FIG. 5.—Mechanical Assembly of Fatigue Machine.


6'
6
4
2
7
1
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9
11
9
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21
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13

FIG. 6.—Mechanical Assembly Drawing.

of contacts in the relay head 6′, which in turn controls the voltage applied to the heating coil of the furnace. The temperature of the mid-point of the specimen is continuously measured by the thermocouple 7, inserted into the $\frac{1}{8}$-in. diameter axial hole in the specimen.

The frame 2 is made of 18-8 stainless steel and is thermally insulated from the main frame 8 by the intermediate insulating blocks 22. Due to this insulation and the fact that 18-8 has a very low thermal conductivity the outflow of heat to the main frame is very small. Maintenance of a constant temperature in the furnace is, therefore, a relatively simple problem.

On the main frame 8 is mounted the reciprocating driving motor consisting of the stator 9, 9, and the armature beam 10 with its punchings 11. The beam 10 is clamped to the torsion bar 12 at the middle while the bar, in turn, is clamped at its ends by the clamp blocks 13. These blocks are attached to the frame 8 through the bar 14 and clamps 15. Lateral motion of the armature beam at the axis of the torsion bar is prevented by the flexible strut 16.

The drive rod 17 connects the lower end of the specimen clamp 3 to the armature beam. Tuning of the system to 120 cycles per second is effected by adjusting the tuning weights 18 and 19, the former for course and the latter for fine adjustments. The three drive coil leads and the two contact leads are brought off through the terminal board 20.

The rubber mountings 21 isolate the 120-cycle vibration to the machine itself so that a minimum of energy is transmitted to the supporting table.

OPERATING PROCEDURE

The procedure followed in setting up the machine for a test is quite simple and has the very great advantage that no accurate alignment of parts is necessary. After the lower clamp is attached the specimen is clamped in the machine and the drive rod then tightened at both ends. The furnace is dropped on and allowed to come up to temperature. If the relay 6 is properly set, 850 F. equilibrium is reached about 90 min. after turning on the furnace power. After the furnace temperature becomes stable, the specimen is tuned to resonance at 120 cycles per second by changing the tuning weights 18 and 19. A line-frequency meter that may be read to 0.02 cycle is used in tuning the system. The amount of weight required to tune to 120 cycles is recorded, for this quantity is used in calculating the maximum stress in the specimen, as will be explained later.

Now the striking of the contacts introduces a non-linear spring constant into the vibrating system, and if the system is driven at a frequency greater than its free natural frequency an instability will set in when the contacts begin to strike. However, if the driving frequency is below the system natural frequency, the system will be quite stable even when the contacts strike. Therefore, before beginning the test enough tuning weight is removed to detune the specimen to about 120.6 cycles so that at all times the driving frequency will be less than the system natural frequency.

The double amplitude of motion is then adjusted to the desired value by observing the target motion in the microscope and simultaneously adjusting the position of the movable contact by turning the crank 23, Fig. 6. This completes the setting-up procedure and no additional attention is required until the specimen fails.

The temperature at the center of the

specimen is continuously recorded on a recording potentiometer. The elapsed cycles are indicated by the movement of a total time meter, shown at the top center of the control cabinet panel, Fig. 3. Upon failure of the specimen there results a reduction in stiffness due to the existence of a crack. As the crack progresses the specimen is detuned to such an extent that the amplitude is reduced and the control contacts cease to strike. When the contacts no longer strike a Thyratron relay opens the main power switch and shuts the machine down, stopping the elapsed-time meter.

CALCULATION OF STRESS

The most obvious procedure by which to obtain the maximum stress in the specimen as a function of the measured double amplitude *d* is as follows: Referring to Fig. 4, remove the drive rod *D*. Then load the lower clamp *C* at the point of drive rod attachment and measure the resulting deflection for various loads. From the load-deflection diagram the moment of the load about the critical section *X-X* as a function of deflection may be computed and from this the stress is easily obtained. However, this apparently simple method is open to three serious objections: (1) at elevated temperatures the deflection under a constant load will vary with time due to creep; (2) the deflection curve for a concentrated loading at the end differs from the actual deflection curve which occurs as the cantilever vibrates in its natural mode; and (3) the use of this method necessitates making at least one load-deflection test for each material and temperature. In view of these objections an alternative method of stress calculation has been developed.

This method consists of calculating the moment diagram due to the inertia loading on the cantilever during vibration and then computing the maximum stress in the specimen from this moment diagram. Knowing the dimensions and the material of all sections of the cantilever (consisting of *A*, *C*, and *H*, Fig. 4), we can calculate (1) the mass distribution and (2) the stiffness distribution along the length. Under the assumption that the cantilever is built-in at the top and knowing the natural frequency from experiment to be 120 cycles per second, the deflection diagram, or normal curve, is graphically determined by the Stodola method.[7] In the course of this determination the natural frequency is obtained. The deviation of this computed natural frequency from 120 cycles is a good measure of the accuracy of the work. From the deflection curve, the mass distribution, and the frequency, the inertia loading diagram is constructed, and then from this loading diagram the moment diagram for unit deflection at the drive rod is computed. Figure 7 (*a*), (*b*), and (*c*) illustrate the cantilever under consideration, the deflection diagram, and the moment diagram, respectively. Using the conven-

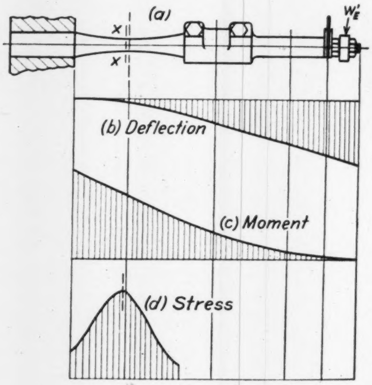

FIG. 7.—Diagrams Used in the Calculation of the Maximum Stress in the Specimen.

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[7] A description of this method may be found in a book by J. P. Den Hartog, "Mechanical Vibrations," First Edition, p. 174, McGraw-Hill Book Co., Inc., New York, N. Y. (1934).

tional formula for the maximum section stress in a beam, $\sigma = \frac{Mc}{I}$, the stress diagram, Fig. 7 (*d*), may be derived from the moment diagram. From this curve it is seen that the critical section *X-X* is slightly removed from the section of minimum diameter, the distance being about $\frac{1}{8}$ in. for the usual specimen.

W_E is the effective weight in grams required to tune the specimen-clamp system to 120 cycles per second.

As a check on this method, load-deflection tests have been made. After correcting for the difference in the deflection diagram between static and dynamic loading, the result checked Eq. 1 to within 1 per cent.

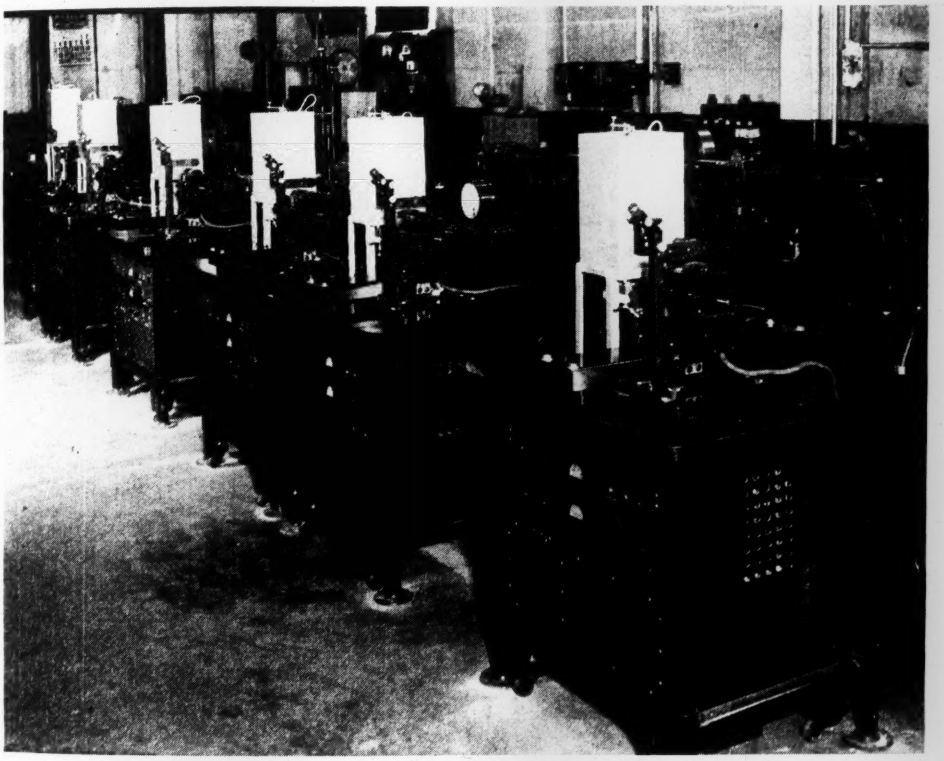

FIG. 8.—View of a Battery of Six Machines Installed in the Laboratory.

In Appendix B[8] it is shown that for a 12 per cent chromium steel specimen at room temperature these calculations give the following formula for the maximum stress in the specimen:

$$\sigma_{cr} = (113{,}700 + 556\ W_E)\ d \quad .\ .(1)$$

where *d* is the double amplitude in inches measured by the microscope, and

Now further considerations have shown that Eq. 1 may be used for other materials provided that the modulus of elasticity does not drop below 20,000,000 psi. and that the density does not differ appreciably from 0.281 lb. per cu. in. A change, say a decrease, in modulus is taken care of in the formula by the W_E term, the weight required to tune the specimen to 120 cycles. The tuning weight, of course, decreases with the modulus. Inasmuch as the effect of elevated temperature on the stress configuration is due almost entirely to the change in modulus of elasticity, Eq. 1 may be also used for high-temperature tests, so that, in general, for any ferrous material and temperature, the stress determination involves only the use of the single formula, Eq. 1.

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[8] See p. 745.

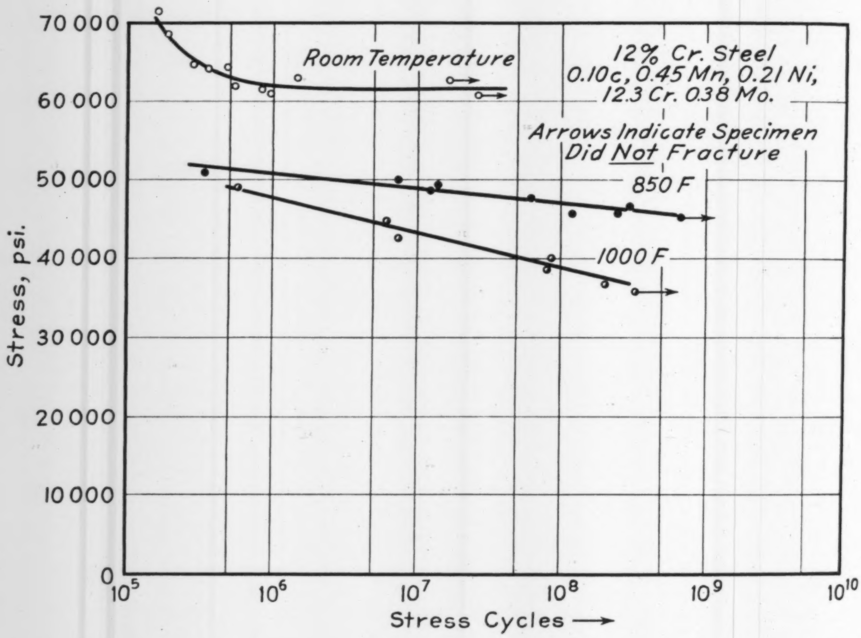

FIG. 9.—*S-N* Diagrams for 12 per cent Chromium Steel at Room Temperature, 850 F., and 1000 F.

An extensive consideration of the various errors involved in this method of stress calculation has shown that the maximum error should be within ± 1½ per cent. It is believed that the largest error involved is that associated with estimating the weights of the various components of the lower clamp but an over-all check of this point was made by weighing the clamp and comparing this weight with the computed weight. The deviation was never greater than 1 per cent.

OPERATING EXPERIENCE AND TEST RESULTS

A battery of six of these machines has been in constant operation at the Westinghouse Research Laboratories for a period of over a year. Figure 8 shows these six machines in the Laboratory. A six-point recording potentiometer continuously records the temperature of each of the specimens under test. Otherwise, each machine is an independent unit.

Fatigue tests have been run on many different types of steels and also on a cast copper alloy. The temperatures at which these tests have been conducted vary from room temperature up to 1000 F. Several specimens have been run out to nearly a billion stress cycles, which requires a running time of about three months.

Figure 9 is a representative *S-N*

diagram showing the results of fatigue tests on a 12 per cent chromium steel at three different temperatures, room temperature, 850 F. and 1000 F. The experimental evidence indicates that at room temperature this steel possesses an endurance limit of 62,000 psi. as is evidenced by the flattening of the *S-N* curve above 2,000,000 cycles. However, at 850 F. and higher temperatures there is no flattening of the curve up to 500,000,000 cycles. Therefore, if we assign a value to the endurance limit, we must also state the corresponding number of cycles at which this value was read off of the *S-N* curve. Thus, we say that at 300,000,000 cycles the "endurance limit" of this steel is 46,000 psi. at 850 F. and 37,000 psi. at 1000 F.

Rotating beam specimens, 0.273 in. in diameter, of this material run at 1725 rpm. gave a room temperature endurance limit of 56,800 psi.

The physical properties of this material are:

Yield point, psi	92 600
Tensile strength, psi	111 000
Elongation, per cent	24
Reduction of area, per cent	67 7
Brinell hardness	228 to 241

Conclusion

The fatigue machine that has been described has been developed to perform the type of tests whose results are presented in Fig. 9. The additional parameter, temperature, and the necessity for running tests out to about 500,000,000 cycles has made a high-speed machine almost imperative for this class of work. This new machine operates at 120 cycles per second, or 7200 cycles per minute. Excepting the fact that the machine is the constant-deflection rather than the constant-load type, all of the desired specifications laid down in the first part of this paper for a high-temperature fatigue tester have been satisfied. In addition, the machine requires no calibration at the various temperatures, and furthermore, there are no bearings or lubrication problems. Six of these machines have been in successful operation in the Laboratory for over a year. They have yielded information which by any other method would have taken a considerably longer time to obtain.

Acknowledgments:

The development of this fatigue machine was a group project in which J. G. Baker, F. C. Rushing, B. D. Cametti, and the authors participated. The fatigue tests shown in Fig. 9 were conducted by P. R. Toolin. The authors wish to thank R. E. Peterson for his help and guidance on this project and L. W. Chubb and the Westinghouse Electric and Manufacturing Co. for permission to publish these results.

APPENDIX A

Description of the Automatic Amplitude Control

The control elements associated with the main mechanism are separately housed, in the black cabinet under the table in Fig. 3. This includes the amplitude control, the furnace temperature control and the automatic shut-off relay. Inasmuch as the temperature control is not particularly novel, consisting only of a temperature-sensitive relay which through other relays puts one or another of two adjustable voltages onto the furnace heating element depending upon whether the furnace is too hot or too cool, the circuit will not be described. A schematic circuit diagram of the amplitude control is shown in Fig. 10. The actual circuit diagram is too involved to reproduce in the limited space herein available. 440-v. three-phase house power is converted to two-phase 110-v. by the Scott connected transformers, *A*. Each of the two phases feeds an identical circuit consisting of a variable auto-transformer, *B*, a pair of a-c. coils on a saturable reactor *C*, and one of the two drive coils *D* on the machine proper. The variable auto-transformers afford a manual control of the voltage available for the reciprocating driving motor, and the reactors are part of an automatic control of the same quantity.

The output current from the power amplifier, shown in Fig. 10, flows in the d-c. coils of the reactors. This current determines the degree of saturation in the reactor and thereby its impedance to the drive-coil current. The amplifier output is controlled by the input voltage which in turn is the output voltage of the stabilizing network shown in Fig. 10. This voltage depends directly upon the intensity of striking of the contacts *L*, *L′*. These contacts are mounted on the armature and the frame of the machine proper. They are shown both in Fig. 4 and Fig. 10 and have been previously described. Since the contacts are in series with the 550 d-c. voltage, their effective resistance determines the current which flows in the network and hence the output voltage. The effective contact resistance depends, of course, upon the intensity with which the contacts strike, and this, for a given contact setting, is a function of the armature amplitude. Thus the impedance of the reactors is controlled by the armature amplitude. The reactor impedance determines in turn the drive-coil current, on which the amplitude depends. The arrangement is such that the reduced effective contact resistance due to increased amplitude increases the (negative) input voltage to the amplifier, thus reducing the amplifier output current. This raises the reactor impedance which results in a reduced drive-coil current. The amplitude is thus reduced which is the correction desired. The function of the resistance-capacitance input network is to stabilize the system and eliminate hunting.

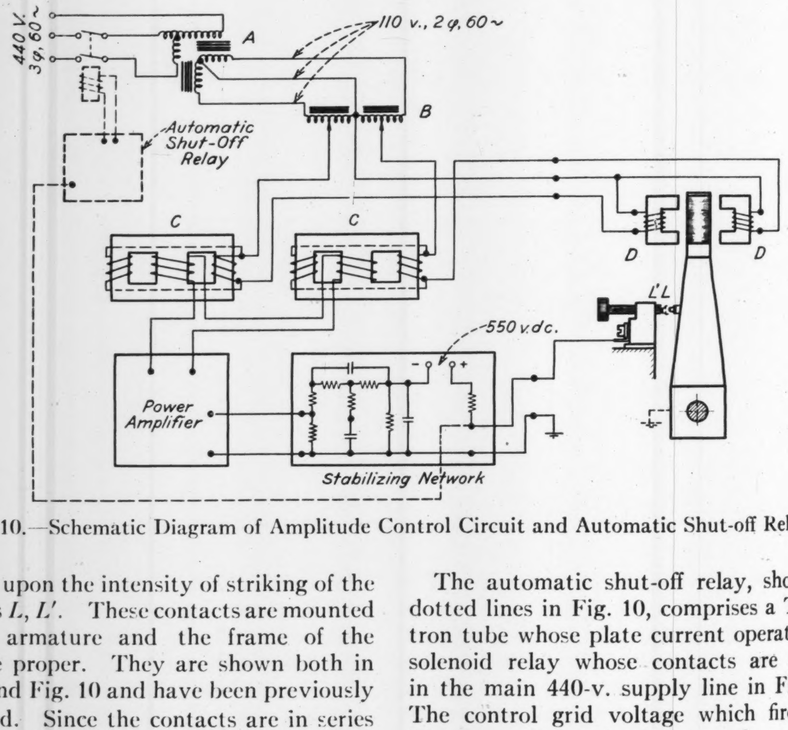

FIG. 10.—Schematic Diagram of Amplitude Control Circuit and Automatic Shut-off Relay.

The automatic shut-off relay, shown in dotted lines in Fig. 10, comprises a Thyratron tube whose plate current operates the solenoid relay whose contacts are shown in the main 440-v. supply line in Fig. 10. The control grid voltage which fires the Thyratron is derived from the voltage across the contacts *L*, *L′*, so that when the contacts are striking the relay is closed, but when the contacts cease striking the relay opens, taking the machine off of the line and at the same time stopping the total time meter. This Thyratron circuit has a delayed action so that the machine does not shut down if the contacts do not strike for one or two cycles.

APPENDIX B

METHOD OF CALCULATING THE MAXIMUM STRESS IN THE SPECIMEN

As was previously discussed in the paper, the deflection and the moment diagrams, Fig. 7 (*b*) and (*c*) are constructed from the mass and stiffness distribution along the cantilever by the graphical method due to Stodola.[7] For this computation a value of the tuning weight necessary to tune the cantilever to 120 cycles, which we shall designate as W_E', was used. In addition, the amplitude at the drive rod, y, was taken as unity. We define the additional symbols:

M_0 = bending moment at the minimum section of the specimen,
d_0 = diameter of minimum specimen,
$M_0 = M_{01} + M_{02}$, where,
M_{01} = bending moment due to mass of specimen and clamp,
M_{02} = bending moment due to tuning weight,
W_E = effective weight of tuning weight, referred to the drive rod;

and, furthermore, when any synbol is "primed," as M_0, the quantity is that for unity amplitude. Let us express the formula for stress in a form in which W_E appears explicitly. It is easy to calculate the moment of the inertia force of W_E about the minimum section. Calling this M_{02}' we have

$$M_0' = M_{01}' + M_{02}' \text{..........} (2)$$

from which M_{01}' may be obtained. Then the total moment for a slightly different value of W_E and for any amplitude y is given by:

$$M_0 = \left(M_{01}' + \frac{W_E}{W_E'} M_{02}'\right) y \text{......} (3)$$

and the maximum stress σ_0 at the section of minimum diameter is

$$\sigma_0 = \frac{M_0}{Z_0} \text{..............} (4)$$

where Z_0 is the section modulus. Now the greatest stress in the specimen, which we shall call σ_{cr}, or the critical stress, does not occur at the minimum section but at a section which is about $\frac{1}{8}$ in. above the minimum section. Knowing the geometry of the specimen (see Fig. 1), the bending moment M_0' and the slope of the moment diagram, $\frac{dM_0'}{dx}$, at the minimum section (see Fig. 7 (*c*)) it is a straightforward problem in calculus to compute the greatest stress in the specimen. The result is:

$$\sigma_{cr} = \left[1 + \frac{Rd_0}{12}\left(\frac{1}{M_0'}\,\frac{dM_0'}{dx}\right)^2\right]\sigma_0 \text{...} (5)$$

R is the radius to which the specimen was machined ($9\frac{1}{4}$ in. in Fig. 1). Combining Eqs. 3, 4, and 5 we obtain the formula for the stress

$$\sigma_{cr} = \left[1 + \frac{Rd_0}{12}\left(\frac{1}{M_0'}\,\frac{dM_0'}{dx}\right)^2\right] \times \left(\frac{32}{\pi d_0^3}\right)\left(M_{01}' + \frac{M_{02}'}{W_E'} W_E\right) y \text{..} (6)$$

in which M_{01} is obtained from Eq. 2 and all other quantities except W_E and y are known. The amplitude y is directly proportional to the double amplitude d measured by the microscope. Substituting the appropriate numerical values into Eq. 6, we obtain the stress formula given in Eq. 1:

$$\sigma_{cr} = (113{,}700 + 556 W_E) d \text{......} (1)$$

DISCUSSION

MR. J. M. LESSELLS.[1]—This is a very welcome addition to the literature of fatigue and describes a very interesting fatigue testing machine.

Test data on the fatigue strength of steels at high temperature are very meager but from what has already been published we generally accept the idea of a drooping curve as shown in Fig. 9.

There is one question: namely, have the authors any explanation for the difference in endurance limits as found by them: namely, 62,000 psi. for the new type of machine and 56,800 psi. for the rotating-beam type?

MR. W. P. WELCH[2] *(author's closure, by letter)*.—Mr. Lessells has asked for an explanation of the difference in endurance limits as determined by this machine and by the rotating-beam machine, an R. R. Moore rotating simple beam machine. This difference may be primarily attributed to the fact that this new machine is a "vibratory" type, while the rotating-beam machine is, as the name implies, a "rotating" type. Endurance limits as determined by vibratory machines are almost universally higher than endurance limits determined by rotating-beam machines, other conditions being the same.

For quantitative data on this subject, let us refer to the current report of the Research Committee on Fatigue of Metals.[3] With this report is included an appendix, entitled, "The Effect of Type of Testing Machine on Fatigue Test Results." As a part of this work, two determinations of endurance limits were made on R. R. Moore rotating simple beam machines and one determination of endurance limit was made on the Krouse mechanical vibratory machine, in which test specimens of the same material and of the same diameter (0.300 in.) were employed. The results of these determinations, for two different steels, is given in the following table:

Material	Endurance Limit, psi.		Ratio: $\frac{(a)}{(b)}$
	(a) Vibratory Type	*(b)* Rotating Type (Mean)	
Heat-treated alloy steel (SAE 4340)	84 500	80 000	1.056
Low alloy steel (HT-50)	47 000	44 000	1.068

The ratio of the endurance limits of the 12 per cent chromium steel, determined by the new machine (vibratory) and the rotating-beam machine, respectively, is 1.084, which is not greatly different from the two ratios reported above. However, it is to be noted that the ratio 1.084 should be corrected for size effect if it is to be directly compared with the committee's results. The diameter of specimen was 0.550 in. for the new machine specimen and 0.273 in. for the rotating-beam machine specimen. The committee's report discussed the size effect as it existed in rotating-beam tests. These results indicate that the endurance limit for a 0.550-in. diameter specimen should be approximately 90 per cent that for a 0.273-in. diameter specimen. However, in correcting our tests for size of specimen it would be necessary to have size effect data determined from vibratory specimens. It is entirely possible that the magnitude of this effect might be considerably different from that determined for rotating-beam specimens.

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[1] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[2] Research Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[3] See p. 132.

TESTING MATERIAL IN THE RESONANCE RANGE

By R. K. Bernhard[1]

Synopsis

The effect of the amplification factor at or near resonance can be used advantageously in testing materials. The fundamental principle governing the investigations at or near resonance are discussed. An oscillator driven endurance test machine based on the resonance principle is described more in detail including control units and calibration. The main purpose of this machine is to test larger specimens and riveted or welded structural units; static and dynamic load conditions, alone or superimposed, can be produced at high frequencies, requiring a rather small power input for the driving motor. A few test data are presented.

Introduction

The evergrowing demand for testing material under combined static and dynamic loads in order to be able to imitate service conditions as closely as possible requires the introduction of new research methods. Those methods which allow investigations under constant and pulsating loads either alone or superimposed are preferable. In Fig. 1 the various combinations of static and dynamic stresses are represented graphically. The two 45-deg. lines indicate the static load variations in the tension or compression range. The sinusoidal curve shows the dynamic load variations, superimposed either as alternating stress in both ranges, tension or compression, or causing a complete stress reversal with no static prestress. Furthermore, larger specimens, or still better, structural units, may replace small test specimens to reduce the often undesirable influence of surface condition, etc. Ordinarily the larger specimens would require a powerful driving mechanism for the testing machine; hence to decrease the power requirements, the amplification factor caused by resonance effects will be of considerable value. However, working in or near the resonance (unstable dynamic equilibrium) demands special controlling devices.

Existing Methods:

Quite a number of testing machines are based on the above mentioned principles. Four characteristic types of machines might be mentioned:

1. Fatigue testing machines with electromagnetic drive.[2]
2. Tension-compression machines driven by mechanical oscillators.[3]

Both types of machines operate at resonance frequencies, but have been built for rather small specimens only.

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[1] Professor, and Head of Department of Engineering Mechanics, The Pennsylvania State College, State College, Pa.

[2] Rayflex Fatigue Testing Machine, *Bulletin 145*, Baldwin-Southwark Co., Philadelphia, Pa.

[3] E. Lehr and W. Prager, "Endurance Testing Machine for Superimposed Alternating Tension-Compression- and Shear-Stresses," *Forschung.*, Vereines Deutscher Ingenieure, Vol. 4, No. 5, September-October, 1933, p. 209.

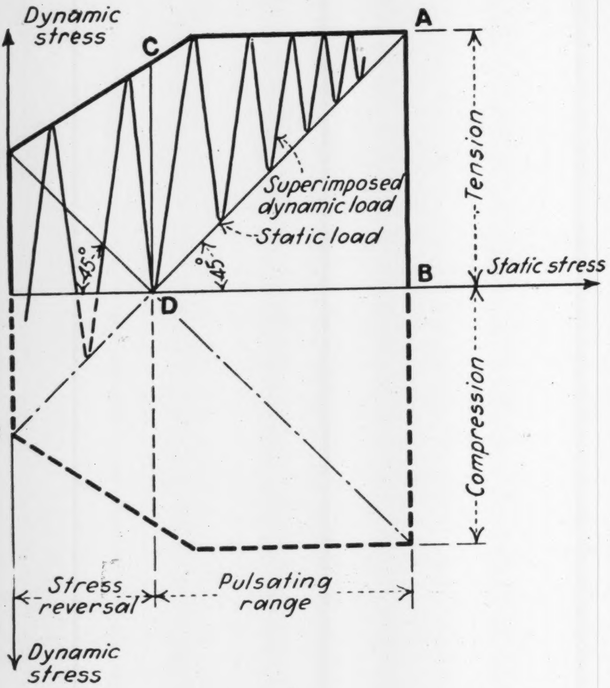
Dynamic stress
C
A
E
Superimposed dynamic load
Static load
Tension
45°
45°
B
Static stress
D
F
Compression
Stress reversal
Pulsating range
Dynamic stress

FIG. 1.—Graphic Representation of Static and Dynamic Stress Ranges.

AB = Static tension only.
CD = Pulsating stress in tension ranges.
EF = Complete stress reversal (tension compression).

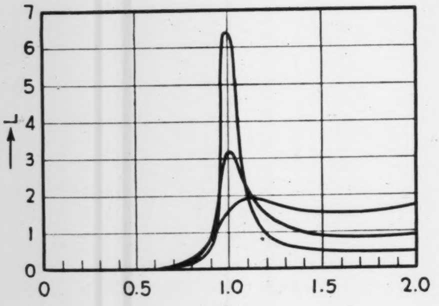

FIG. 2.—Resonance Curves for Exciting Force Increasing with the Square of the Frequency (ω^2_e), Power (L), Discord (Z) Curves.

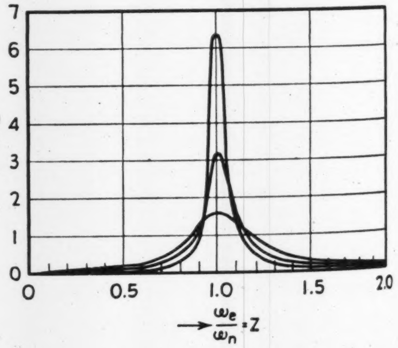

FIG. 3.—Resonance Curves for Constant Exciting Force, Power (L), Discord (Z) Curves.

Notes for Figs. 2 and 3

The X-axis represents the ratio of the exciting frequency to the natural frequency, called discord or tuning factor

$$\left(Z = \frac{\omega_e}{\omega_n}\right).$$

On the Y-axis the corresponding power input (L) is plotted. The three curves in each figure demonstrate the influence of different damping coefficients ($\alpha_1 = 0.1, \alpha_2 = 0.2$, and $\alpha_3 = 0.4$).

3. Fatigue testing machines with eccentric drive.[4]

4. Pulsator types, driven by hydraulic means (oil pumps).[5]

These two latter types can be used for larger specimens; however, they operate substantially below the resonance frequencies, thus, are confined to rather low frequencies.

Hence, none of these machines fulfill all three conditions simultaneously, namely, (1) are able to test large specimens or structural units, (2) reduce the power input by working in or near resonance, and (3) allow higher working frequencies.

Difficulties Involved:

The purpose of this paper is to discuss the previously mentioned difficulties for this type of testing and to describe a machine where a satisfactory control at or near the resonance frequency has been achieved. To keep the resonance range under control, three specific considerations are important:

1. The unstable dynamic equilibrium near the resonance point represents the most important consideration as can be seen from the power-discord curves of a one degree of freedom system. In Figs. 2 and 3, two characteristic families of response curves[6] for different damping values are shown. The discord (z), that is, the ratio of exciting angular frequency (ω_e) to natural angular frequency (ω_n) is plotted against the power input (L). In Fig. 2 forces increasing as the square of the exciting frequency and, in Fig. 3 constant exciting forces (independent of the frequency) are assumed.

All curves indicate that in the resonance range a small change in discord corresponds to a large difference in power input. Furthermore, for exciting forces increasing as the square of the frequency (Fig. 2), the larger damping causes the maximum values of the power L to shift towards higher discords. Only with constant exciting forces, as shown in Fig. 3, can this shift in the resonance peak, caused by larger damping values, be avoided, thus yielding curves which are almost symmetrical with respect to the vertical axis at $z = 1$.

Hence the first step is to stabilize the location of the resonance peak. This can be achieved by some means which will allow the excitation of vibration with a constant force amplitude independent of the discord.

2. The second difficulty is caused by the steep incline of the curves on either side of the resonance point. This can be overcome by electrical means. To stabilize the driving motor, that is, to cause it to keep a constant frequency independent of the load, a Ward-Leonard motor generator set with an adjustable diverter across the generator series field can be used. By proper adjustment of a compound shunt, the motor will remain always at the required frequency regardless of whether the steep upward portion of the response curves causes an abrupt increase, or the downward portion an abrupt decrease in the required power input.

3. Finally the third step consists of a continuous automatic control of both the desired static load and the superimposed dynamic load, either alone or combined. Mechanical devices can be used advantageously, such as adjustable maximum and minimum contacts; these contacts are energized by superfrequency apparatus, as for example seismic pendulums which respond to changes (relative motion) only, either of the desired constant

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[4] W. W. Wilson, "Fatigue Tests of Riveted Joints," *Civil Engineering*, Vol. 8, No. 8, August, 1938, p. 563.

R. L. Templin, "Fatigue Machines for Testing Structural Units," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 711 (1939).

[5] "Universal Testing Machine," *Zeitschrift* des Vereines Deutscher Ingenieure, Vol. 80, No. 47, November, 1936, p. 1404.

Otto Graf, "On Endurance Experiments with Flat Bars and Riveted Joints of Light Metals," *Der Stahlbau*, Vol. 8, August 16, 1935, p. 132.

[6] W. Späth, "Theory and Application of Oscillators," Julius Springer, Berlin (1934).

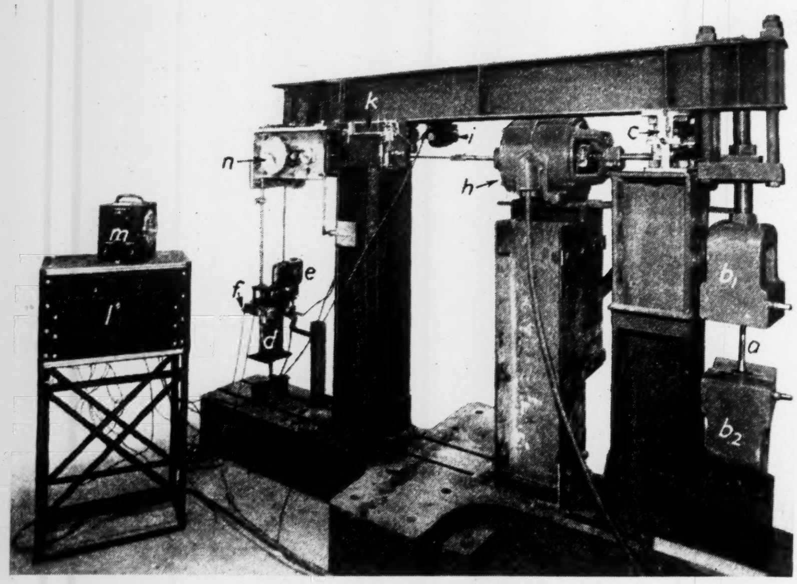
k
i
c
n
h
m
l'
e
f
d
b₁
a
b₂

FIG. 4.—Endurance Testing Machine for Static Tension or Compression and Superimposed Pulsating Stresses.

a = Specimen.
b_1 and b_2 = Grips.
c = Flexible leaf joint.
d = Main spring for static prestress.
e = Prestress control motor.
f = Prestress control pendulum.

g = Mechanical oscillator to excite alternating stresses.
h = Oscillator motor.
i = Alternating stress control motor.
k = Alternating stress control pendulum.
l = Manual control box for alternating stress and prestress.
m = Stroboscope.
n = Phasemeter.

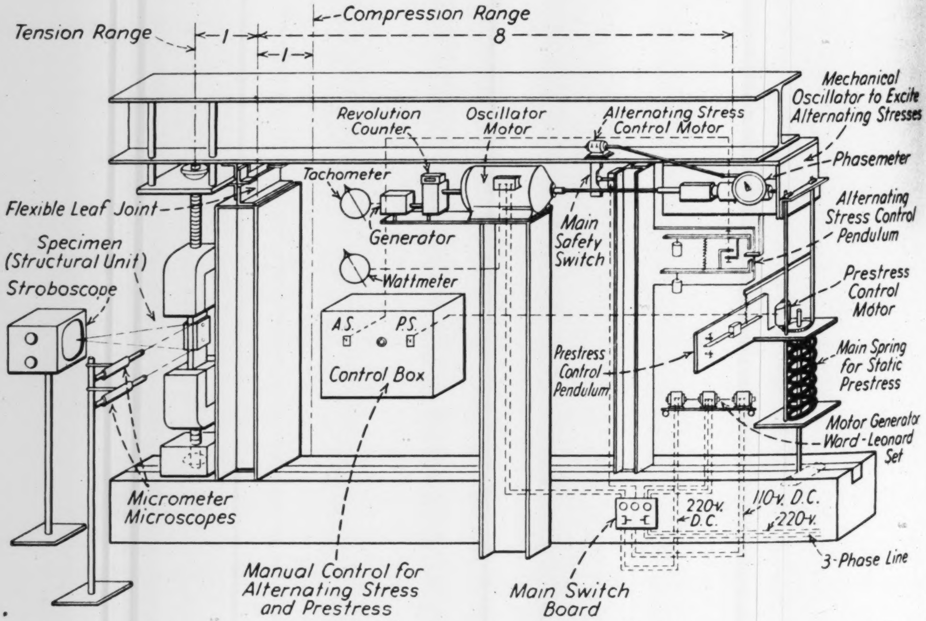
Compression Range
Tension Range
1
1
8
Mechanical Oscillator to Excite Alternating Stresses
Revolution Counter
Oscillator Motor
Alternating Stress Control Motor
Phasemeter
Tachometer
Flexible Leaf Joint
Generator
Main Safety Switch
Alternating Stress Control Pendulum
Specimen (Structural Unit)
Wattmeter
Stroboscope
Prestress Control Motor
A.S.
P.S.
Control Box
Prestress Control Pendulum
Main Spring for Static Prestress
Motor Generator Ward-Leonard Set
Micrometer Microscopes
220-v. D.C.
110-v. D.C.
220-v.
3-Phase Line
Manual Control for Alternating Stress and Prestress
Main Switch Board

FIG. 5.—Endurance-Testing Machine. Diagram of mechanical set-up including control units.

prestress or the desired alternating stresses.

ENDURANCE TESTING MACHINE

The testing machine (Fig. 4) as developed in the laboratory for testing materials of the Pennsylvania State College (School of Engineering) is based on the above principles. An endeavor is made to overcome most of the disadvantages associated with tests performed near or in the resonance range. Furthermore the machine permits investigations of large specimens or structural units. Finally, research including combinations from the tension to the compression range upon which are superimposed sinusoidal alternating stresses (see Fig. 1) are made possible.

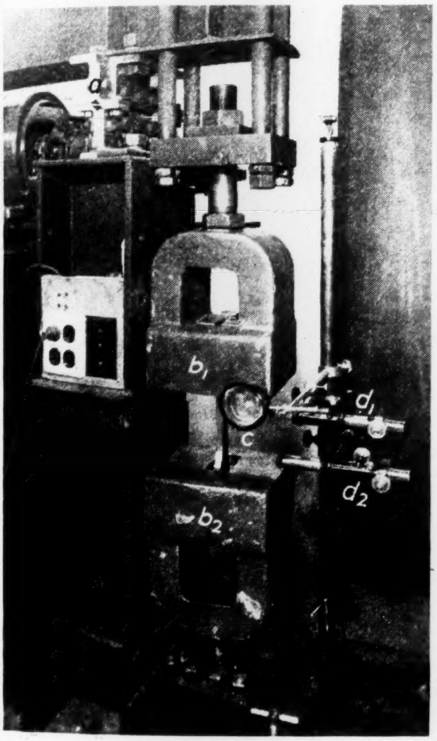

FIG. 6.—Flexible Leaf Joint (*a*), Grips (b_1 and b_2), Specimen (*c*) and Cathetometer (micrometer-microscopes d_1 and d_2).

The general setup is shown schematically in Fig. 5. The cantilever beam with a lever ratio of 1:8 transmits the required stresses to the test piece. A frictionless steel leaf joint (Fig. 6) supports this beam.

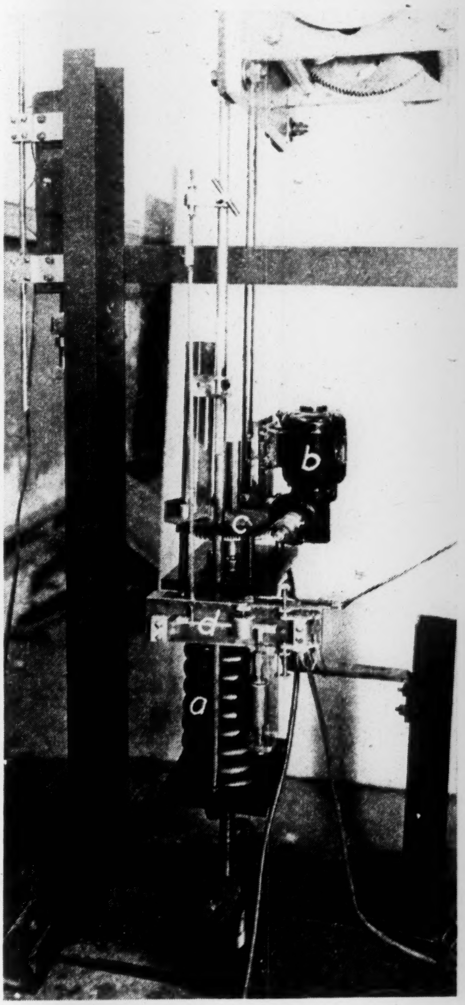

FIG. 7.—Static Prestress- or Precompression Control.

a = Spring.
b = Control motor.
c = Worm gear drive.
d = Control pendulum.

Static Prestress:

Interchangeable calibrated compression springs (Fig. 7) are used to stress the specimen either in static tension or compression, depending upon whether the specimen is fixed beyond or between the flexible joint and the interchangeable spring.

The spring constant of these springs governs to a considerable extent the natural frequency of the complete vibrating system for vertical displacements. By proper selection of the stiffness of these springs the required resonance frequency can be obtained.

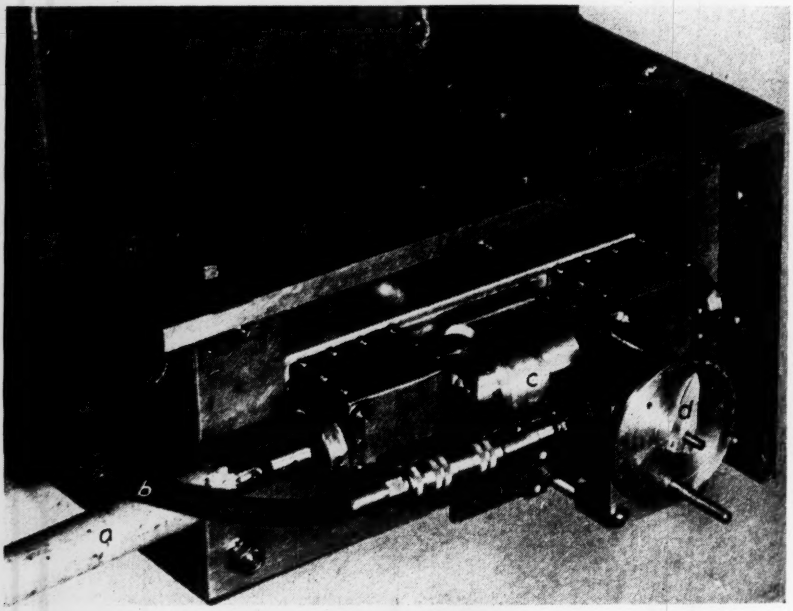

FIG. 8.—Mechanical Oscillator.

a = Shaft to main oscillator driving motor.
b = Flexible shaft to eccentricity control motor.
c = Differential gear arrangement for eccentricity control.
d = Eccentricity indicator.

Dynamic Superimposed Stress:

A mechanical oscillator (Fig. 8) excites the superimposed dynamic stresses.[7] The oscillator consists of two pairs of eccentrically supported masses. Both pairs are rotating in opposite directions in such a manner that only the vertical components of their centrifugal forces remain. A differential gear arrangement allows a change of eccentricity of the rotating system while the machine is in operation (see Figs. 4 and 5). Thus the first requirement of a constant force amplitude, that is, symmetrical power-frequency response curves as shown in Fig. 3, is fulfilled.

CONTROL

Static Prestress:

The main prestress spring is compressed for the required amount by means of a worm gear drive, connected with a small reversible electric motor (Fig. 7). This motor is controlled with a low frequency pendulum, damped by a double acting air dash pot. The upper end of the pendulum is rigidly fixed to the adjustable end of the main prestress spring. The lower end of the pendulum does not participate in the vibratory motion and stands still with respect to the mean position of the main spring (see Fig. 7). Adjustable maximum and minimum contacts are energized by the horizontal arm of this pendulum which controls the motion of motor attached to the fixed end of the main spring. Thus the compression of the main spring and hence the prestress of the specimen is automatically held constant at the required amount.

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[7] R. K. Bernhard, "Dynamic Tests by Means of Induced Vibrations," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 634 (1937).

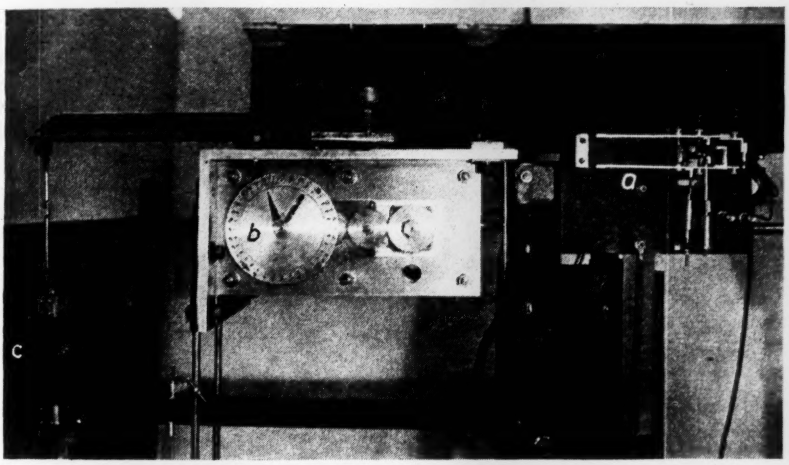

FIG. 9.—Dynamic Stress Control.

a = Double pendulum.
b = Phasemeter.
c = Zero point contact for b.

Superimposed Dynamic Stress:

Frequency.—As mentioned previously, a Ward-Leonard motor generator set with an adjustable diverter supplies the direct current for the driving motor ($\frac{1}{2}$ hp.) of the oscillator. Hence neither an increase nor a decrease in load, even in the resonance range, will change the working frequency of the testing machine. Thus the second requirement, a constant operating frequency, is fulfilled.

Amplitude.—The amplitudes of the pulsating stress are controlled in a manner similar to that used for controlling the static prestress. A small reversible motor automatically changes the eccentricity of the two rotating pairs of weights of the oscillator, hence adjusts the magnitude of the vertical component of the excited centrifugal forces. A low frequency double pendulum arranged in a horizontal position controls by means of adjustable maximum and minimum contacts the magnitude of the alternating stress amplitude (Fig. 9). The double pendulum is attached to the nonmovable part of the testing machine. Each individual arm of the pendulum is damped by a separate air dash pot acting in one direction only. A short lever transmits the displacements of the main cantilever arm to the horizontal pendulums. Any increase of the displacement-amplitude of the main system causes a spread of the two horizontal pendulums, thus energizing the corresponding maximum contact. This contact controls the rotation of the reversible motor in such a way as to reduce the eccentricity of the oscillator and hence the displacement amplitude. For any decrease of the displacement-amplitude, the opposite procedure takes place.

The wiring diagram in Fig. 10 shows the general electrical setup and in particular the arrangement of the four amplifying tubes in the control box. These tubes, controlled by the two pairs of adjustable contacts (maximum or minimum) described above, energize the relays for the two motors; the first motor governs the prestress and the second motor the displacement-amplitude. Thus the third requirement, the rigid control of the static and dynamic loads is fulfilled.

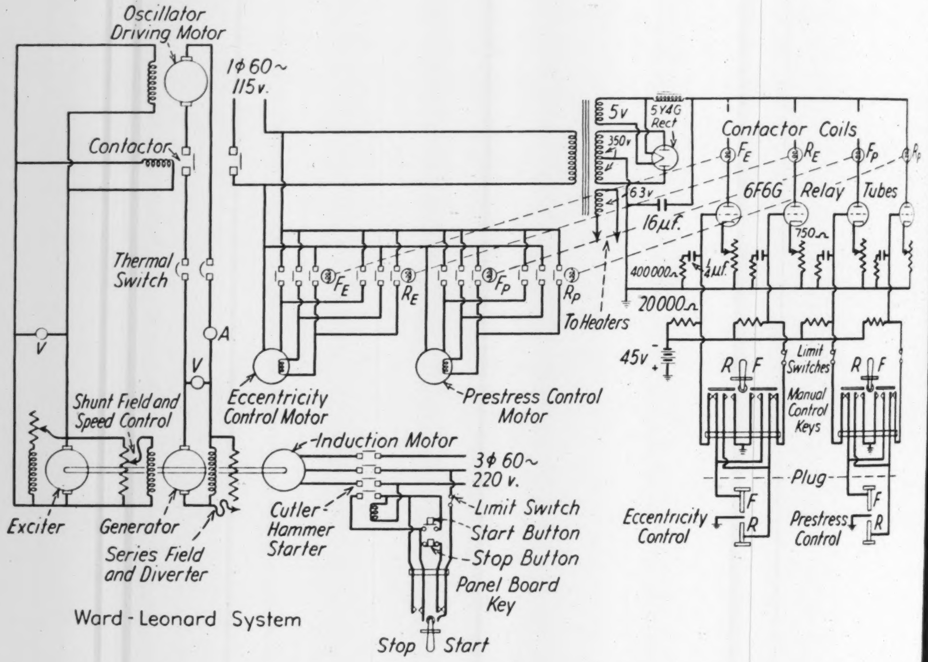
Oscillator Driving Motor
1φ 60~
115v.
Contactor
Thermal Switch
V
A
V
Shunt Field and Speed Control
Exciter
Generator
Series Field and Diverter
Ward-Leonard System
5v
5Y4G Rect
350v
6.3v
16μf.
Contactor Coils
F_E
R_E
F_P
R_P
6F6G Relay Tubes
750Ω
400000Ω
$\frac{1}{4}$μf.
20000Ω
To Heaters
45v
F_E
R_E
F_P
R_P
Eccentricity Control Motor
Prestress Control Motor
Induction Motor
3φ 60~
220 v.
Cutler-Hammer Starter
Limit Switch
Start Button
Stop Button
Panel Board Key
Stop
Start
R F
Limit Switches
Manual Control Keys
R F
Plug
F
R
Eccentricity Control
F
R
Prestress Control

FIG. 10.—Endurance-Testing Machine. Electric-wiring diagram.

A cathetometer (see Fig. 6) permits an independent check whether the calibrated spring as well as the calibrated oscillator transmits the required stresses to the specimen or structural unit. Furthermore, undesirable eccentricities or slippage caused by the gripping of the specimen can be controlled. The cathetometer consists of two micrometer microscopes with a sensitivity of 2 × 10^{-6} in. Any motion of two minute fiduciary marks attached directly on the structural unit in a distance corresponding to the desired gage lengths may be measured by these two micrometer microscopes. The two fiduciary marks can be illuminated by a stroboscope, tuned-in to the working frequency of the machine, hence making possible the taking of readings at any time during the test procedure. The change in distance of these fiduciary marks indicates directly the actual deformation (strain or stress) in the specimen or structural unit.

Furthermore a phasemeter can be

used to determine the damping and reveals the smallest deviations from any established dynamic equilibrium. Phase deviations might be caused by minute fatigue cracks in the specimen not even visible with a microscope. The phasemeter consists of a lever repeating the position of the rotatable weights of the oscillator (see Fig. 9). This lever revolves within an angular ring graduated in degrees. Independently a contact is made whenever the main cantilever arm of the testing machine passes the zero line (mean position), energizing a flash of the stroboscope. These stroboscope flashes illuminate both rotating lever arm and scale on the angular ring. The illuminated lever points to zero phase when the motion of the oscillator masses and the main cantilever coincide. A phase difference of 90 deg. indicates resonance regardless of the amount of damping. The phasemeter reading is one of the most sensitive means of indicating changes in the dynamic equilibrium (stable stage) thus making possible the close observation of progressive fatigue failures.

Adjustable maximum and minimum switches automatically prevent any static and dynamic overloads; a revolution counter indicates the number of cycles endured by the specimen or structural unit.

Finally a displacement-amplitude meter, a tachometer (Fig. 11), and a wattmeter are necessary to plot response curves as shown in Figs. 2 and 3.

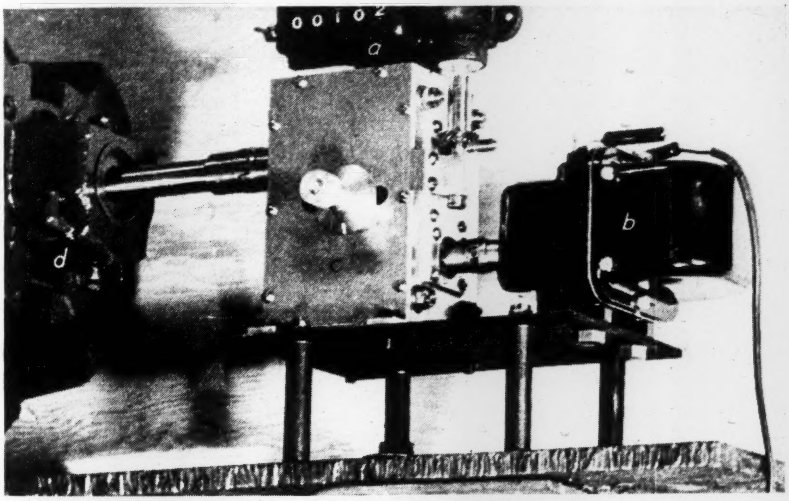

FIG. 11.—Impulse and Frequency Control.

a = Revolution counter.
b = Generator for tachometer.
c = Variable gear drive between oscillator motor and *b*.
d = Main oscillator motor.

The following data represent test results secured by means of this machine:

In Fig. 12 (*a*) to (*c*), three types of families of response curves are plotted. All curves refer to the same specimen made of mild steel with 0.76 sq. in. cross-section and 8 in. length between the grips. On the X-axis the exciting frequencies are plotted; on the Y-axis the corresponding values are shown, in Fig. 12 (*a*) for the stress amplitude in the specimens, in Fig. 12 (*b*) for the power input of the oscillator motor, and in Fig. 12 (*c*) for the phase angle between force and displacement vector. The different curves of each figure indicate three stages, first, the initial setup, second, the beginning of slip or a fatigue failure and third, a progressive fatigue failure.

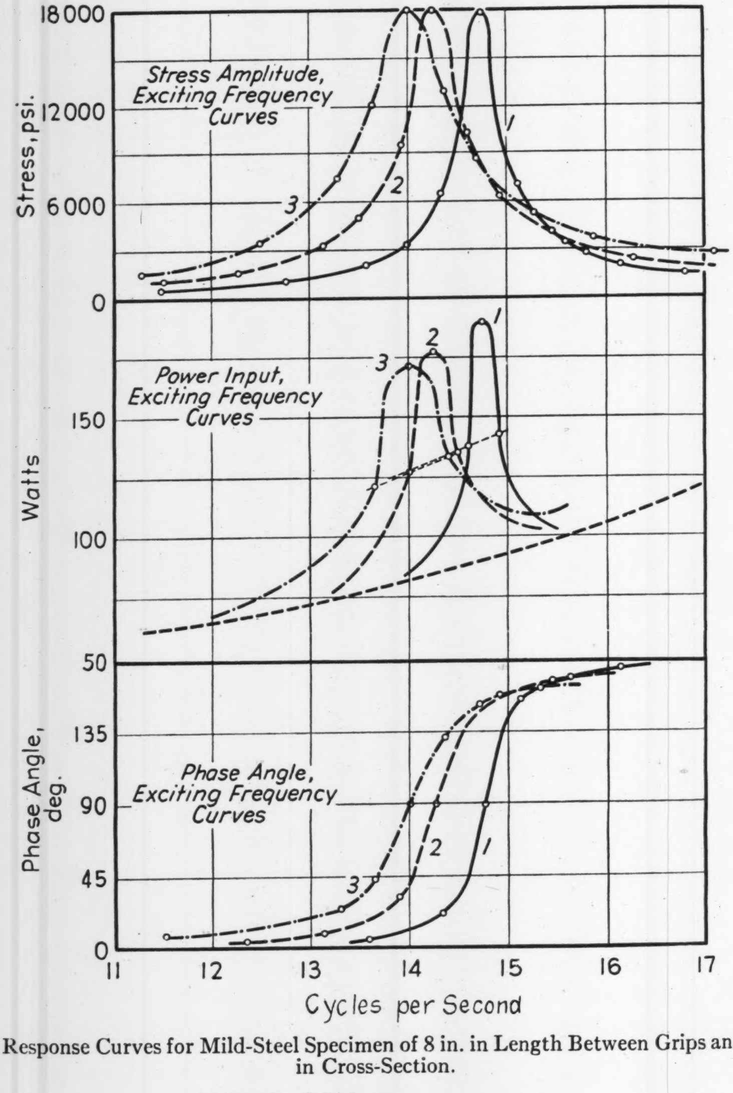

FIG. 12.—Response Curves for Mild-Steel Specimen of 8 in. in Length Between Grips and 0.76 sq. in. in Cross-Section.

Curve 1 = Initial setup.
Curve 2 = Beginning of fatigue failure.
Curve 3 = Progressive fatigue failure.

The curves of Fig. 12 (*a*) and (*b*) indicate, through progressive testing, the drop of the exciting frequency at which the maximum stress or maximum power input occurs. Since these maxima almost coincide with the resonance frequency, the spring constant of the vibrating system must have decreased likewise. Furthermore, the broadening of the individual loops from curves 1 to 3 shows a pronounced increase in damping.

This growth in damping may also be seen clearly in Fig. 12 (*c*) as causing the decreasing slope in the phase angle-frequency curves from 1 to 3. All three curves in Fig. 12 (*c*) pass the 90-deg. line of the phase angle at the corresponding resonance points as shown in Fig. 12 (*a*) and (*b*).

The tests were carried out under the following conditions:

Maximum static working load (constant prestress)	±40 000 lb.
Maximum dynamic working load (alternating stress)	±50 000 lb.
Maximum working frequency	1 500 cpm.

For mild steel specimens with 0.76 sq. in. cross-section and 8 in. gage length:

Damping = 0.063
Amplification factor = 50

S-N diagrams of smaller mild steel specimens did not show any difference as compared with results obtained on ordinary fatigue machines.

The resonance frequency can be varied from approximately 500 to 1500 cycles per minute. The required natural frequency may be obtained by selecting a main spring with the proper spring constant.

The fact that with a small motor of only $\frac{1}{2}$ hp. the theoretical dynamic force excited at resonance in a specimen or structural unit will approach 800,000 lb. (maximum capacity of oscillator × lever ratio × amplification factor = 2000 × 8 × 50), appears to the author as opening up a field of investigation offering new possibilities.

The author takes this opportunity to acknowledge the valuable help of Messrs. K. de Juhasz, E. Axman and H. N. Shoiket in designing the machine.

AN ACCELERATED ATMOSPHERIC CORROSION TEST

By H. Pray[1] and J. L. Gregg[2]

Synopsis

As a part of an extended investigation of the atmospheric corrosion resistance of ordinary and low-alloy steels, a test procedure and equipment have been developed which are capable of predicting in a few weeks the relative atmospheric corrosion rates for materials of this type. The paper includes a general discussion of atmospheric corrosion testing, a description of the procedure and apparatus used for the accelerated test, and a comparison of the accelerated and actual atmospheric tests for a number of commercial and special steels.

The complete evaluation of a new construction material for outdoor use requires a knowledge of its atmospheric corrosion behavior. This may be of minor engineering importance if suitable coatings are applied and maintained, although if the material is intentionally used or unintentionally becomes bare, reliable information on resistance to atmospheric attack is indispensable.

Any comprehensive research program aimed at the development of a new steel for outdoor use, whether new in composition or in method of processing, must, therefore, include some determination of atmospheric corrosion resistance. Atmospheric exposure tests, to be significant, require many specimens exposed in a variety of atmospheres over a period of several years. These requirements of atmospheric exposure testing have been recognized by this Society and many other official agencies.

The usual methods of atmospheric exposure testing result in a corrosion rate - time curve, which for ordinary and low-alloy steels show that the rate decreases with increasing time of exposure. The rate of corrosion for a steel exposed for a given period is primarily dependent upon the degree of atmospheric pollution and the moisture conditions. These obviously vary with location and with the season at which exposure is started. In view of the dependence of rate upon duration of exposure and upon the vagaries of atmospheric conditions, it is impossible quantitatively to define a value which is entirely characteristic of the atmospheric corrosion behavior of a steel. Further, a single type of atmospheric exposure test specimen yields rate values that may differ considerably from those of actual service.

However, the relative atmospheric corrosion rates for a group of steels, if sufficiently comprehensive with respect to the number of samples, the duration, and the locations of exposure, constitute engineering information of value in the selection of materials. The relative corrosion rates for ordinary and low-alloy steels after a few months exposure bear no relation whatever to their relative rates at times corresponding to those of expected service. Short-time atmospheric exposure tests therefore are not only valueless, but can be dangerously misleading. The relative rates for a group of materials of the class under discussion also vary with the exposure location. This variation, although real, is in general much less than might be expected.

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In spite of the limitations in the results of atmospheric exposure testing, it is evident that a means of securing atmospheric relative rate data without the tedious and time-consuming procedure ordinarily involved would be an extremely useful tool. The necessity of waiting several years before the effect of a composition or processing variable becomes apparent, constitutes a serious deterrent to the development of alloys for which a reasonable knowledge of atmospheric corrosion resistance is required for adequate appraisal. The final answer to any corrosion problem must admittedly be based on actual service behavior. An accelerated or pilot test is valuable only to the extent that it serves to hasten and simplify the arrival at that goal.

A successful accelerated test must be capable of producing relative rate values (for a range of material composition within the scope of possible interest) which are within the spread of the relative rate values disclosed by field tests. It must show good correlation with actual long-time exposure tests before it can be used with confidence.

The development of the test to be described is based upon laboratory and field investigations of the atmospheric corrosion of low-alloy steels, including a study of the effects of atmospheric sulfur dioxide, type, frequency, and duration of wetting, etc., which have extended over a period of years and which will be discussed elsewhere. Since the primary purpose here is to describe the accelerated test apparatus and method rather than the differences in atmospheric corrosion resistance of steels resulting from variations in composition, only that portion of the data necessary to establish the validity of the test will be cited.

APPARATUS AND TEST PROCEDURE

The apparatus is shown diagrammatically and in part in Fig. 1. It consists essentially of a revolving specimen rack with a capacity of 96 specimens so mounted that the specimens travel through three compartments. The largest of these is a high humidity thermostat, the temperature of which is regulated by the temperature of a layer of water beneath the specimens. Humidity, air circulation, and water circulation are maintained by pumping the water at a controlled temperature through a nozzle so placed in the free space in the center of the specimen rack and directed downward that the water does not strike the samples. The compartment is also provided with baffled sprays to supplement the dew formation that occurs on the samples and to provide means of securing intermittent runoff.

A small glass-walled compartment is used for drying. Air is forced upward through it by the blower shown in the diagram. The air temperature is controlled by resistance heaters in the air duct.

Refrigeration is accomplished in the adjoining compartment, in which the samples pass close to the walls of a metal jacket through which precooled liquid is circulated.

The specimens are sheet tension test specimens, 0.75 in. by 12 in. by No. 22 gage. They are suspended rigidly on stainless steel wires between the upper

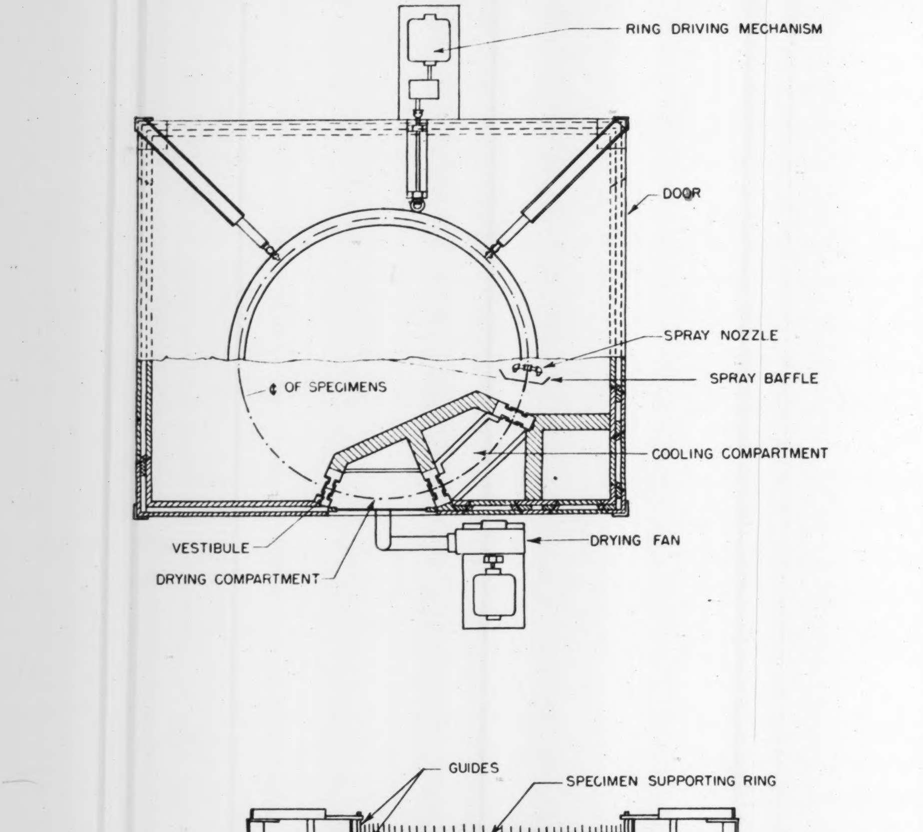
RING DRIVING MECHANISM
DOOR
SPRAY NOZZLE
SPRAY BAFFLE
¢ OF SPECIMENS
COOLING COMPARTMENT
VESTIBULE
DRYING FAN
DRYING COMPARTMENT
GUIDES
SPECIMEN SUPPORTING RING
WATER SEAL
SPECIMENS
BRINE COOLING TANK
COOLING COIL
BRINE
REFRIGERATION UNIT
BRINE CIRCULATING PUMP

Fig 1.—Corrosion Testing Apparatus.

and lower rings of the specimen carrier in a position normal to the radius of the rings. The openings between adjacent compartments are thus very narrow. The atmosphere from one compartment is prevented from entering the next compartment by maintaining a slightly reduced pressure in the small vestibules that are provided between them.

The space beneath the three compartments is used to house the operating equipment, namely, the compressor, thermostat, and circulating pump for the refrigerating liquid, air blower for the drying compartment, pump for the circulating water in the high-humidity compartment, controls for the sprays, and suction pump for the vestibules.

With this apparatus it is possible to attain complete uniformity of treatment of the individual samples, a wide range of wetting conditions, controlled temperature of the three phases of the cycle, and complete independence of the wetting, drying, and cooling conditions. As a result of numerous tests under varying conditions of operation, the following were satisfactory:

Rate.—Three revolutions per hour. This gives three complete cycles of 15 min. wet, 2.5 min. drying and 2.5 min. cooling per hour.

Drying.—The blower rate and air temperature are adjusted so that corroded samples appear dry at a point about half way through the drying compartment. This results in a sample temperature of approximately 110 F. on leaving this compartment.

Cooling.—The temperature of the cooling liquid is adjusted to 38 F. At this coolant temperature the specimen temperature as it leaves the cooling compartment is approximately 55 F. It was found that the specimen temperature at this phase is not critical, the principal function of the compartment being to bring the specimen temperature somewhat below that of the wetting compartment.

Wetting.—The temperature of the wetting compartment is maintained at 110 F. The amount of water on the specimen appears to be the most critical factor in the entire cycle. Dew formation resulting from the entrance of a cold specimen into the high-humidity compartment is not sufficient. It is necessary to use intermittent baffled sprays, controlled by a solenoid valve operated by a timing mechanism, in order to get uniform wetting just short of run-off. It is further necessary to spray continuously at a rate sufficiently high to give active run-off for a period of three cycles per 24 hr. The purpose of this is to remove soluble corrosion products.

As will be shown later, the above-described procedure alone is entirely incapable of reproducing the relative atmospheric corrosion rates of low-alloy steels. No manipulation of the various phases of the cycle has been found that will duplicate atmospheric exposure results on bare steel. The latter appears to be highly dependent upon the condition of the initial corrosion film, which film is not reproduced by the cycle described. If, however, the initial film is formed by an actual atmospheric exposure as brief as 1 month, the cycle will then duplicate the later stages and will give, in the course of a 2-weeks test, relative rates that are comparable with those of extended atmospheric results.

The complete test then resolves itself into a short (1 month) atmospheric exposure, which has been found to be independent of the season within ordinary limits, followed by a 2-week exposure to the above test cycle. The combined pre-exposure and indoor test thus requires 6 weeks for completion and yields relative rates equivalent to several years of outdoor exposure.

MATERIALS

Test results are presented for the three series of steels listed in Table I. All materials were rolled to No. 22 gage (approximately 0.032 in.), box annealed at 1650 F., and pickled in hydrochloric acid prior to either atmospheric or accelerated testing. The tension data in the table were obtained with standard sheet tension test specimens. Analyses are ladle analyses. The steels of series *A* were known to show a wide variation in atmospheric corrosion resistance and were used as a yardstick in the development of the test. The atmospheric data for series *B* and *C* were secured subsequent to the accelerated test data. The latter, therefore, constitute predictions later verified by the atmospheric tests.

TABLE I.—COMPOSITION AND TENSILE PROPERTIES.

Steel	Composition, per cent								Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium	Copper			
SERIES *A*											
E	0.03	0.04	0.012	0.030	0.01	0.05	....	0.08	35 470	46 800	30.5
B	0.09	0.51	0.050	0.028	0.05	....	....	0.07	25 150	41 650	33.0
D	0.10	0.48	0.045	0.025	0.05	....	....	0.25	27 400	46 600	31.0
M	0.09	0.52	0.167	....	0.69	....	0.99	0.50	42 650	66 200	23.5
P	0.07	0.38	....	....	....	....	2.00	0.30	23 400	46 750	24.0
Q	0.09	0.46	....	....	....	....	4.76	0.31	29 450	55 550	23.0
SERIES *B*											
CV	0.10	0.51	0.02	0.043	0.13	....	....	....	29 100	45 550	30.0
GA	0.11	0.53	....	0.11	0.14	....	....	....	28 600	45 700	27.0
GB	0.11	0.48	....	0.17	0.12	(no Al added)			30 700	49 050	25.0
GC	0.11	0.51	....	0.16	0.18				29 500	45 800	25.5
SERIES *C*											
JG	0.11	0.64	0.100	0.04	0.31	0.76	0.61	0.68	52 600	70 600	26.5
JH	0.10	0.65	0.100	0.03	0.38	0.81	0.62	0.61	54 000	73 200	25.0
JN	0.10	0.59	0.16	0.03	0.36	1.01	0.64	0.67	57 500	77 300	27.0
JR	0.11	0.71	0.150	0.04	0.39	1.98	0.61	0.61	65 500	82 000	25.0
JT	0.12	0.70	0.060	0.04	0.41	3.04	0.61	0.61	64 400	85 100	23.5
JV	0.12	0.77	0.14	0.03	0.41	2.99	0.61	0.60	69 400	91 900	23.5
KG	0.08	0.38	0.008	0.038	0.025	....	....	....	25 700	41 300	38.0
D	....	....	0.045	....	....	0.25					

TEST RESULTS

Atmospheric Tests:

Sheet tension test specimens were exposed in racks identical with those designed and used by the A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. The racks were located at Bethlehem on the roof of the main office building of the Bethlehem Steel Co., a typically industrial location; at Columbus, on the roof at the Battelle Memorial Institute in a typically residential section; at Boston near salt water; and at Pittsburgh, near the river in a highly polluted industrial atmosphere. Duplicate samples were removed periodically, stripped of rust by cathodic treatment in sodium cyanide solution and weight losses determined. A sufficient number of samples was exposed initially so that this procedure could be carried on over a period of years, or until complete failure of the sheet sample occurred.

The relative rate data for the three series, calculated from the weight loss per unit area, are contained in Table II. The weight loss data for the steels of series *A* exposed at Columbus are the

TABLE II.—RELATIVE ATMOSPHERIC CORROSION RATES.[a]

Steel	1 yr.				2 yr.				3 yr.				4 yr.			
	(1)	(2)	(3)	(4)	(1)	(2)	(3)	(4)	(1)	(2)	(3)	(4)	(1)	(2)	(3)	(4)
Series *A* (Rate for Steel D = 100)																
E	150	157	148	143	151	163	151	147	162	165	150	154	169	172	152	172
B	114	118	106	106	112	113	108	115	116	114	106	110	111	113	108	113
D	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
M	77	68	76	79	63	60	67	62	54	53	61	52	55	49	58	50
P	94	81	93	93	80	73	84	81	73	69	77	68	74	67	75	72
Q	28	28	42	33	26	25	37	26	24	23	33	23	22	21	30	21
SERIES *B* (RATE FOR STEEL CV = 100)																
CV	100	....	100	100	100	....	100	100	100	....	100	100	100	....	100	100
GA	119	....	120	110	144	....	138	149	164	....	142	....	148	....	149	150
GB	136	....	135	118	234	....	208	174	200+	....	197	....	....	....	188	200+
GC	151	....	136	147	282	....	182	182	200+	....	190	....	....	....	178	200+
SERIES *C* (RATE FOR STEEL D = 100)																
JG	84	....	82	74	75	....	70	57	61	....	59	55				
JH	86	....	86	75	75	....	73	58	61	....	61	56				
JN	77	....	76	67	66	....	65	53	53	....	53	52				
JR	77	....	74	63	61	....	62	52	50	....	51	49				
JT	74	....	66	60	59	....	56	50	50	....	46	45				
JV	65	....	64	53	52	....	53	41	44	....	46	40				
KG	163	....	134	159	181	....	139	148	199	....	141	163				
D	100	....	100	100	100	....	100	100	100	....	100	100				

[a] (1) = Bethlehem. (2) = Boston. (3) = Columbus. (4) = Pittsburgh.

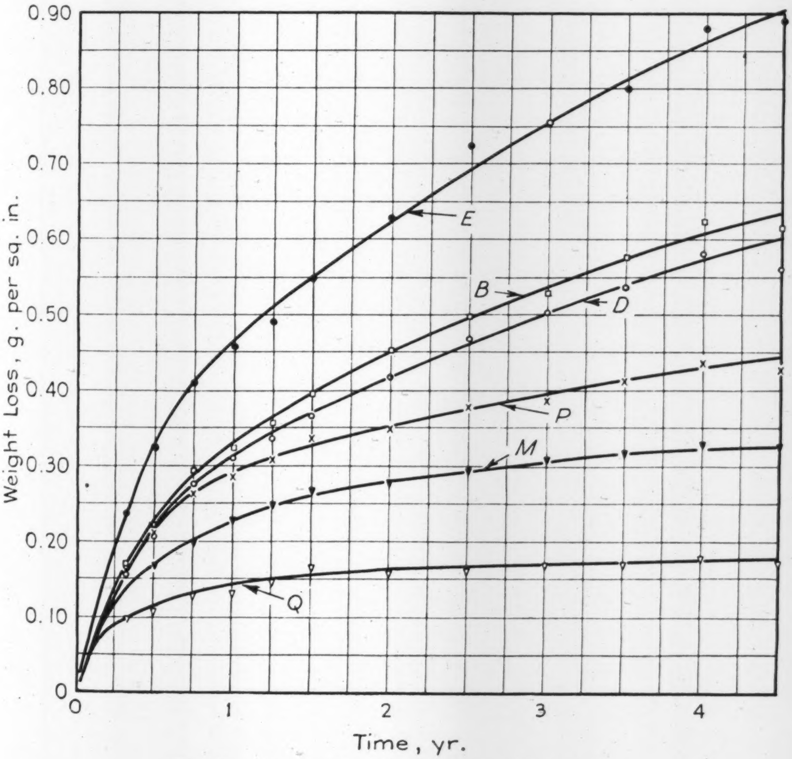

FIG. 2.—Atmospheric Corrosion at Columbus.

basis for the corrosion-time curves of Fig. 2.

The curves show clearly that the corrosion rates change considerably with time, and, since the rate of change is dependent upon steel composition, the relative rates may also be expected to change with duration of exposure.

According to the data of Table II, the change in average relative rate at the several locations with time after a 1-yr. period is appreciable. Thus, steel P would be rated as less superior to D after a 1-yr. exposure than after a 4-yr. exposure. For periods shorter than 1 yr. the relative corrosion rates are very different from those determined after longer periods of exposure.

TABLE III.—COMPARISON OF ACCELERATED TEST WITH ATMOSPHERIC EXPOSURE DATA.

Mark	Atmospheric 1 yr.			Atmospheric 2 yr.			Atmospheric 4 yr.			One Month Atmospheric	One Month Indoor Cycle	Accelerated Test[a]	
	Max.	Min.	Avg.	Max.	Min.	Avg.	Max.	Min.	Avg.			No. 1	No. 2
						SERIES *A*							
E	157	143	150	163	151	153	172	152	164	106	160	143	133
B	118	106	111	115	108	112	113	108	111	102	117	103	110
D	100	100	100	100	100	100	100	100	100	100	100	100	100
M	79	68	75	67	60	62	58	49	54	100	140	66	60
P	94	81	90	84	73	80	75	67	72	106	175	83	82
Q	42	28	33	37	25	29	30	21	24	100	94	61	53
						SERIES *B*							
CV	100	100	100	100	100	100	100	100	100	100	....	100	100
GA	121	110	117	144	138	141	....	....	....	106	....	128	139
GB	136	118	130	234	174	205	....	....	....	100	....	135	140
GC	150	136	144	282	182	215	....	....	....	100	....	147	147
						SERIES *C*							
JG	84	74	80	75	57	67	....	....	....	....	....	72	....
JH	86	75	82	75	58	65	....	....	....	....	....	71	....
JN	67	77	70	66	53	61	....	....	....	....	....	74	....
JR	77	63	71	62	52	58	....	....	....	....	....	68	....
JT	74	60	67	59	50	55	....	....	....	....	....	55	....
JV	65	53	61	53	41	48	....	....	....	....	....	47	....
KG	163	134	152	189	139	158	....	....	....	....	....	183	....
D	100	100	100	100	100	100	....	....	....	....	....	100	....

[a] One month atmospheric plus 2 weeks indoor cycle.

Considering again the average relative rates at the several locations, the atmospheric data are reasonably consistent in rating the steels of a given series. In series *A*, steel Q is outstandingly superior and steel E inferior. After 1-, 2-, 3-, or 4-yr. exposure, the relative rates are invariably in the order, Q, M, P, D, B, and E.

The relative rates for a single material and exposure period exhibit a certain variation with the location of the test racks. In spite of the variety of atmospheres represented, the spread in relative rate values is not great enough to alter the order of corrosion resistance of the steels at the different locations. To reproduce relative rate figures within this spread and, therefore, to predict the relative atmospheric corrosion resistance for similar series of steels in a shorter time than is required for atmospheric exposure, is the function of the accelerated test.

Accelerated Tests:

Table III lists the maximum, minimum, and average atmospheric relative rates observed at the various stations after 1, 2, and 4 yr. of outdoor exposure. These constitute a summary of the data of Table II. It also lists the relative rates observed after exposure at Columbus for 1 month, the relative rates shown by a 1-month exposure to the accelerated cycle without prior atmospheric exposure, and the relative rates resulting from the combined 1-month atmospheric plus 2-week indoor cycle previously described. For series *A* and *B* the results of duplicate accelerated tests are given.

The 1-month atmospheric results show no correlation with the relative corrodibility of the various steels, as revealed by more extended atmospheric exposure. All of these steels appear to corrode at approximately the same initial rate.

The results of exposure to the laboratory test without first permitting the specimens to acquire an initial atmospheric corrosion film are obviously meaningless.

In general, the relative rates shown by the accelerated test, which comprises a 1-month atmospheric exposure plus a 2-week laboratory exposure, fall within the maximum and minimum atmospheric values after an outdoor exposure of from one to four (roughly two) years. This is the case for all of the materials listed in Table III, with the exception of the relatively highly resistant steel Q.

The order of corrodibility of steels of any of the three series is as clearly revealed by the accelerated test data as by the long-time atmospheric exposure tests at the several test stations. In series *C* there may be some question as to the accelerated test value for steel JN, but it is apparent that the differences in atmospheric corrosion resistance of steels JG, JH, and JN are small and not extremely significant, as shown by either accelerated or atmospheric tests.

CONCLUSIONS

It is possible to evaluate the atmospheric corrosion behavior of steels in 6 weeks with the same degree of reliability as can be obtained by extended atmospheric exposure. The test procedure involves the formation by nature of an initial rust film as indoor conditions have not been found that duplicate this initial film. The specimens are then subjected to a controlled laboratory cycle, which apparently reproduces the active conditions (such as when wet with dew) of atmospheric corrosion with the omission of the long, nearly inactive (dry and completely wet) periods that naturally exist.

The steels may vary in composition and corrosion resistance over a considerable range. The results constitute a prediction of sufficient accuracy to be useful. From the many steels tested (data for which are not recorded here) no significantly misleading results have been secured and the method has proven itself useful in the development of low-alloy steels in so far as the evaluation of the effect of alloy additions and combinations thereof on atmospheric corrodibility is concerned.

The accelerated test is not presented as applicable to the determination of the atmospheric behavior of all materials, nor as a possible standard atmospheric test for low-alloy steels. It is presented as an example of the fact that short-time tests for specific purposes can be devised and can be used to eliminate in part the necessity for tedious and time-consuming field tests.

Acknowledgments:

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AN EQUATION REPRESENTING THE RATE OF DEVELOPMENT OF RUST ON GALVANIZED IRON SHEETS AS ESTIMATED BY THE A.S.T.M. TEST

By J. B. Austin[1]

Synopsis

An equation has been derived which represents, within the accuracy of observation, the change of rate of rusting of galvanized iron sheets with change in environment and change in weight of zinc coating as estimated by the tests carried out by Subcommittee VIII of Committee A-5 at Altoona, Pa., Brunot Island (Pittsburgh), Pa., Sandy Hook, N. J., and State College, Pa. The equation does not represent the data obtained at Key West, Fla. This relation besides suggesting a simple method of plotting the data in order to obtain a straight line, which type of plot has advantages over the usual graph, also enables one to derive an empirical number which can be used as an index of the corrosiveness of a given atmosphere to galvanized iron sheets when the test is made by the A.S.T.M. procedure. If the observed rate of rusting can be represented by the equation, this index can be evaluated from data for the sheets with the thinnest coating of zinc, which rust the most rapidly, and can then be used to estimate the rate of development of rust on sheets with a heavier coating.

The influence of environment and of weight of zinc coating on the rate of development of rust on galvanized iron sheets has been studied at five locations over a period of many years by the Society's Committee A-5 on Corrosion of Iron and Steel.[2] The results show that at each of the four northern stations, namely, Altoona, Pa., Brunot Island (Pittsburgh), Pa., State College, Pa., and Sandy Hook, N. J., the average rate for a given class of sheet is represented by an S-shape curve of the type shown in Fig. 1. As a large number of observations are required to determine each curve and as the test must be carried on over many years, particularly for the sheets with the heaviest coatings, it would be helpful to establish a general pattern of behavior which would enable a prediction, on the basis of less extensive observations, of the continued rate of development of rust on heavily-coated sheet from the more rapid rate on lightly-coated material. Moreover, it is convenient to have a method of plotting the data which yields a straight line, since this greatly reduces the number of observations required to determine the position of the curve, interpolation is simplified, extrapolation can be carried out with greater assurance, and occasional erratic measurements are readily recognizable by their deviation from the best line through the whole set of points.

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The rate curve as ordinarily drawn (Fig. 1) resembles certain curves which have been widely studied, notably, the rate curve for an autocatalytic reaction, the rate of isothermal transformation of austenite in steel, and the ogive curve, which suggests that the data for the rusting of galvanized sheets might be correlated by means of established relations representing one of these several curves. Of these relations, that representing the rate of an autocatalytic chemical reaction is the simplest and has, chiefly for this reason, been used in the present study.

Direct test using data published in 1935 by the Society[2] shows that by a slight modification of the autocatalytic equation it is possible to obtain, for the observations taken at the four northern stations, a simple relation which adequately expresses the change of rate of rusting with environment and with weight of zinc coating. This equation, besides suggesting a simple method of plotting the data in order to obtain a straight line, also enables the derivation of an empirical number, which can be used as an index of the corrosiveness of each atmosphere to galvanized iron sheets when the test is made by the A.S.T.M. procedure.[3] This does not, of course, imply that the rate of development of rust on galvanized sheets is an autocatalytic reaction; indeed, the applicability of the equation implies nothing

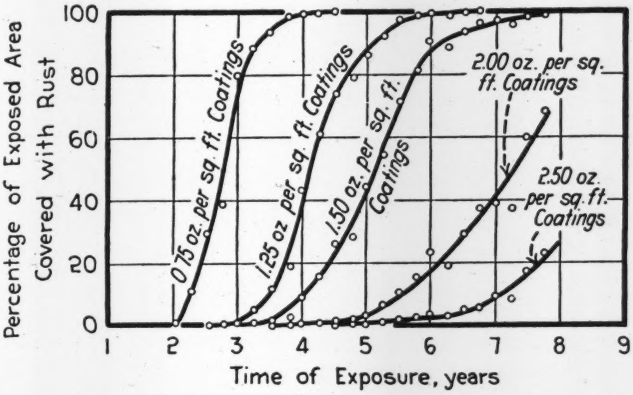

FIG. 1.—Rate of Development of Rust on Galvanized Iron Sheets Exposed at Brunot Island.

TABLE I.—VALUES OF LOG (P/100 − P) FOR INTEGRAL VALUES OF P.

P	0	1	2	3	4	5	6	7	8	9
0	∞	−2.00	−1.690	−1.509	−1.380	−1.276	−1.195	−1.125	−1.060	−1.005
10	−0.954	−0.907	−0.866	−0.824	−0.788	−0.754	−0.721	−0.688	−0.658	−0.630
20	−0.602	−0.575	−0.550	−0.524	−0.500	−0.478	−0.455	−0.432	−0.410	−0.389
30	−0.368	−0.348	−0.327	−0.308	−0.288	−0.269	−0.250	−0.231	−0.212	−0.194
40	−0.176	−0.158	−0.140	−0.123	−0.105	−0.087	−0.070	−0.052	−0.035	−0.017
50	0.000	0.017	0.035	0.052	0.070	0.087	0.105	0.123	0.140	0.158
60	0.176	0.194	0.212	0.231	0.250	0.268	0.288	0.308	0.327	0.348
70	0.368	0.389	0.410	0.432	0.454	0.477	0.500	0.525	0.550	0.575
80	0.602	0.630	0.659	0.689	0.720	0.753	0.788	0.826	0.865	0.908
90	0.954	1.005	1.060	1.125	1.195	1.276	1.380	1.509	1.690	2.000

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[2] Report of Subcommittee VIII, of Committee A-5, on Field Tests of Metallic Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 93 (1935); Vol. 38, Part I, p. 88 (1938).

[3] Report of Subcommittee VIII, of Committee A-5, on Field Tests of Metallic Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 191 (1927).

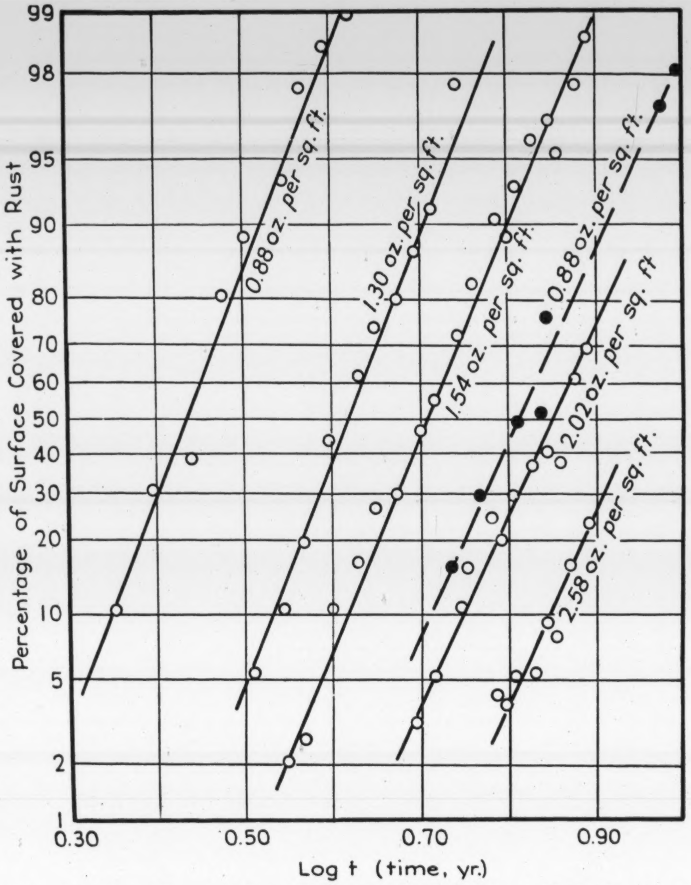

FIG. 2.—Chart Showing Rate Curves for Brunot Island (Full Lines) and for Sandy Hook (Dashed Lines) Plotted with the Autocatalytic Scale as Ordinate and a Logarithmic Scale of Time as Abscissa. Coating weights are actual averages.

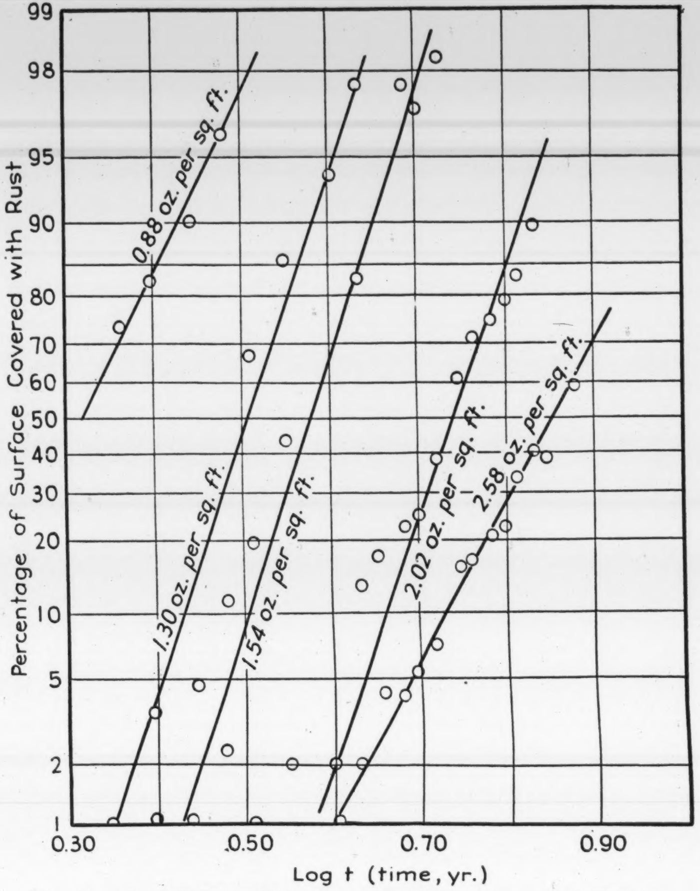

FIG. 3.—Chart Showing Rate Curves for Sheets Exposed at Altoona Plotted with the Autocatalytic Scale as Ordinate and the Logarithmic Scale for Time as Abscissa. Coating weights are actual averages.

whatever as to the mechanism of rusting but merely demonstrates empirically that the mathematical expression derived for the one case holds for the other.

The equation for the rate of an autocatalytic reaction can be written

$$\log\left(\frac{P}{100 - P}\right) = k_1 + k_2 t \ldots\ldots(1)$$

where P is the per cent transformed in time t, and k_1 and k_2 are constants. If a set of measurements follows this equation, they yield a straight line when log $(P/100 - P)$ is plotted against t. A plot of this sort is easily drawn with the help of Table I, which gives values of log $(P/100 - P)$ for integral values of P. A graph of this kind, can, however, be constructed without the calculation of log $(P/100 - P)$ by using as one coordinate the autocatalytic or logistic grid suggested by E. B. Wilson.[4] This is simply a scale in which the interval between any two values of P is proportional to the difference between the corresponding values of log $(P/100 - P)$. With such a scale on one axis and a linear time scale on the other, a straight line is obtained when observed values of P are plotted directly against t.

The use of Eq. 1 does not, however, give a straight line with the A.S.T.M. corrosion data since the autocatalytic curve is symmetrical in time about the point $P = 50$, whereas the observed curve for the rate of development of rust is not. Thus, on the curve given by Eq. 1, the time required for the reaction to go from 50 per cent transformation to completion is the same as that required to arrive at 50 per cent transformation from the beginning, whereas the typical curves given in Fig. 1 show that the time required for zinc to disappear from a surface half covered with rust is considerably greater than the interval between the appearance of rust and 50 per cent coverage. This difference indicates that in deriving an equation for the observed rate of rusting it is necessary to replace the linear time scale by a scale based on some other function.

A satisfactory function is the logarithm, so that if log t be substituted for t in Eq. 1, the resulting equation

$$\log \frac{P}{100 - P} = k_1 + k_2 \log t \ldots\ldots(2)$$

fits the experimental data surprisingly well, as is shown by the straight lines in Figs. 2 and 3 in which the A.S.T.M. measurements[5] are plotted using the autocatalytic scale as ordinate and the logarithm of time as abscissa. It is interesting to note that Eq. 2 also represents the rate of isothermal transformation in steel with fair accuracy[6] and has been shown by Avrami[7] to be a good approximation to the equation expressing the rate of a reaction which occurs by the formation and subsequent growth of nuclei.

The advantage of the straight lines shown in Figs. 2 and 3 over the original S-shape curves in extrapolation and in smoothing the observations is evident. For example, the development of rust on the material with a 2.5-oz. coating exposed at Brunot Island (see Fig. 1) is quite slow, and it will clearly take many years for the surface to become completely covered with rust, so that if one has to depend solely on observation the time required for complete coverage cannot be determined for some time to come. Yet from extrapolation of the straight line in Fig. 2, it can be said with reasonable certainty that this class of sheet will on the average be 80 per cent covered with rust in 10 yr. and will be 99 per cent covered in 13.5 yr.

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[4] E. B. Wilson, "The Logistic or Autocatalytic Grid," *Proceedings*, Nat. Academy Sciences, Vol. 11, p. 451 (1925).

[5] I am indebted to C. D. Hocker for the numerical values corresponding to the data given in the published curves.

[6] J. B. Austin and R. L. Rickett, "Kinetics of the Decomposition of Austenite at Constant Temperature," *Metals Technology*, September, 1938, T.P. 964.

[7] Melvin Avrami, "Kinetics of Phase Change—II," *Journal of Chemical Physics*, Vol. 8, p. 212 (1940).

The most surprising feature in Figs. 2 and 3, however, is not the fact that a straight line results in each case but that all of the lines have substantially the same slope. The lines in Fig. 2 are all parallel within the error of observation and although the slope of the lines of Fig. 3 is not so uniform as in Fig. 2, the variation is nevertheless not large; moreover, in no case does the slope of the curves in Fig. 3 differ markedly from that in Fig. 2.

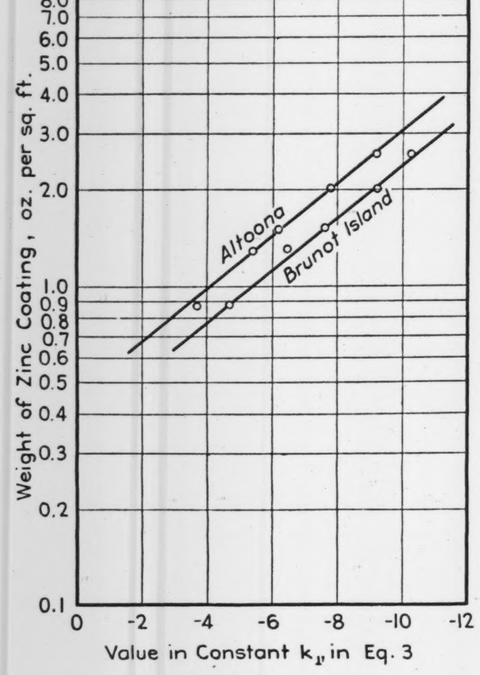

FIG. 4.—Chart Showing Variation of Constant k_1 (Eq. 3) with Weight of Coating for Two Stations.

It appears, therefore, that the slope of the rate line on such a graph is the same for all three locations and all weights of coating tested, which means that the constant k_2 in Eq. 2 has a single value for all the sheets studied. The value of k_2 is about 10.7, hence, within the accuracy of the estimates

$$\log (P/100 - P) = k_1 + 10.7 \log t \quad (3)$$

The data also reveal another significant, though less evident, regularity, namely, that the separation of the curves for different coatings is very nearly the same in both Fig. 2 and Fig. 3, which implies that the relative position of the several lines is determined by the relative weight of coating. This means that the constant k_1 in Eq. 2 is a function of the weight of coating. This function is again logarithmic, as is shown by Fig. 4 in which is plotted the weight of coating on a logarithmic scale against the value of k_1 for the curves for Altoona and Brunot Island on a linear scale.

In drawing these lines the actual average weight of coating[8] has been used instead of the nominal weight for each class of sheet. It will be noticed that the curves for the two stations are parallel, which indicates that the change in k_1 with weight of coating is the same for each atmosphere. On the basis of these curves, again within the accuracy of the estimates,

$$k_1 = -(a + 12.3 \log w)$$

where w is the average actual weight of coating expressed in ounces per square foot and a is a constant which depends upon the location of the test and can therefore be used to characterize the corrosiveness of each atmosphere to galvanized iron sheets as determined by the A.S.T.M. test procedure.[3] For this reason, it will be referred to as the "atmosphere index." Combining Eq. 4 with Eq. 3 we have

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[8] The weight of zinc coating for sheet is always expressed in ounces per square foot of sheet, which, since the sheet is coated on two sides, is twice the average weight of coating per square foot of surface. That is, a nominal coating of 1.0 oz. per sq. ft. means that there is a total of 1 oz. of zinc on the front and back surfaces of each square foot of sheet. The average weight of coating per square foot of each surface is therefore 0.5 oz.

$$\log (P/100 - P) = -a - 12.3 \log w + 10.7 \log t \quad (5)$$

which gives the influence of type of atmosphere, weight of coating, and time on the development of rust under the conditions specified above; and once the value of a is known, the rate curves for that location can be calculated with reasonable accuracy. This is illustrated by Fig. 5 which compares the values calculated by means of Eq. 5 (dashed lines) with the A.S.T.M. observations (circles) for Altoona and Brunot Island. The "atmosphere index" for the several stations at which the A.S.T.M. observations were made is given in Table II.

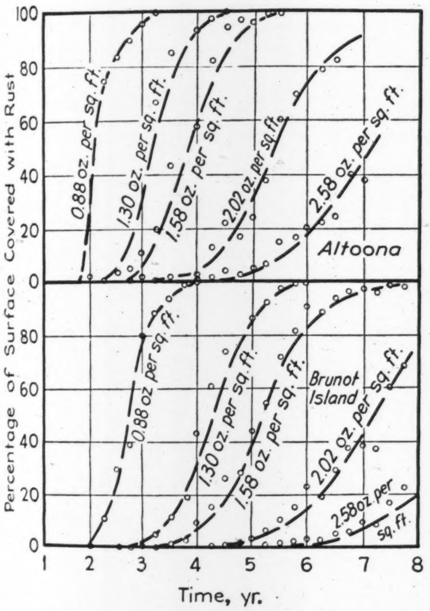

FIG. 5.—Comparison of Rate Calculated by Means of Eq. 5 (Dashed Lines) with A.S.T.M. Observation (Circles) for Two Stations.

TABLE II.—ATMOSPHERE INDEX (a) FOR DIFFERENT STATIONS.

Altoona, Pa.	4.1
Brunot Island (Pittsburgh), Pa.	5.4
Sandy Hook, N. J.	9.6
State College, Pa.	13.6

The practical usefulness of Eq. 5 may be illustrated by the following example:

Suppose that a number of galvanized iron sheets bearing different weights of coating are put out at a new station for test by the A.S.T.M. procedure. The first sheets to rust will be those with the thinnest coating, and a relatively few observations on these rapidly-rusting sheets suffice to show whether or not Eq. 5 is applicable since, if the observations give a straight line when plotted on logarithmic-autocatalytic coordinates, it is reasonably safe to assume that the equation holds. These relatively few observations also suffice for the determination of the index a, since the weight of coating w and the percentage coverage P for several values of elapsed time t are known. Once a is determined, Eq. 5 enables a reasonably accurate calculation of the variation of P with t for any weight of coating w. To take a specific instance, the rate of development of rust at the State College station[9] is extremely slow; thus, the sheets with a nominal coating of 0.75 oz., which class shows the most rapid rusting of those tested, had less than 10 per cent rust after nearly 12 yr., so that a very long time will be required to obtain complete measurements on all weights of coating But the few observations now at hand permit a reasonably satisfactory determination of the index a in Eq. 5 and from this the curve for each weight of coating can be calculated. As an aid in calculations of this sort curves are given in Figs. 6 and 7, respectively, showing the variation of 10.7 log t with t and of 12.3 log w with w.

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[9] Report of Subcommittee VIII, Committee A-5, on Field Tests of Metallic Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 84 (1938).

In most applications it is desired to know the time required for the initial appearance of rust and for this purpose Eq. 5 can be put into a somewhat simpler form. The time of initial appearance of rust (t_1) is commonly, though quite arbitrarily, taken as the time for 1 per cent coverage, hence log $(P/100 - P) = -2.00$ and

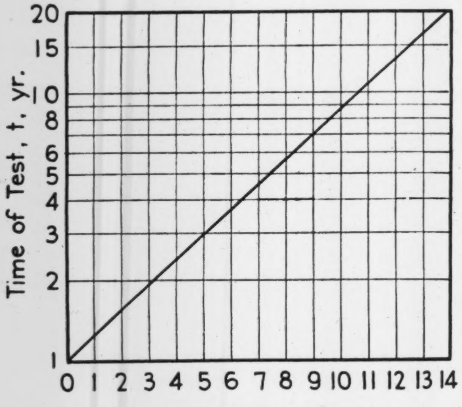

FIG. 6.—Chart Showing Variation of 10.7 log t with t for Use with Eq. 5.

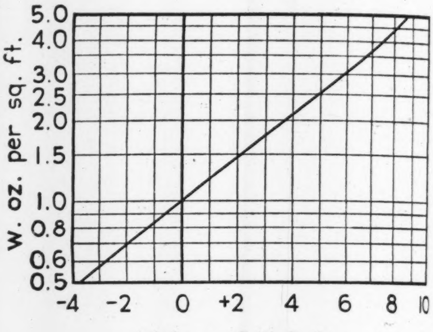

FIG. 7.—Chart Showing Variation of 12.3 log w with w for Use with Eq. 5.

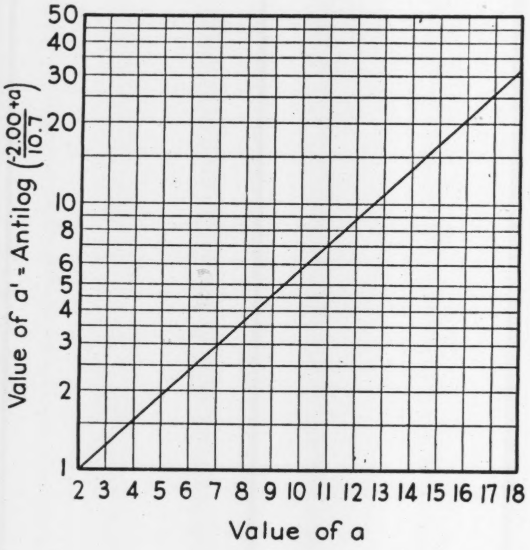

FIG. 8.—Variation of $a' = \text{antilog}\left(\frac{-2.00 + a}{10.7}\right)$ with a for use with Eq. 9.

$$\log t_1 = \frac{-2.00 + a}{10.7} + 1.15 \log w \ldots (6)$$

or

$$t_1 = \left[\text{antilog}\left(\frac{-2.00 + a}{10.7}\right)\right] w^{1.15} \ldots (7)$$

Since the antilog factor is constant for a given atmosphere, Eq. 7 may be written

$$t_1 = a'w^{1.15} \ldots\ldots\ldots (8)$$

The value of a' may be determined conveniently from a by means of Fig. 8. The exponent of w is so nearly equal to unity that no great error is introduced if it is made unity, in fact the equation

$$t_1 = a'w \qquad (9)$$

seems to fit the observations somewhat better than Eq. 8. The values calculated from Eq. 9 are compared in Table III with the A.S.T.M. estimates as published.[9]

A word of warning should be given against extrapolating to $w = 0$, that is to black plate. The index a applies only to galvanized sheet and does not characterize the corrosiveness of a given atmosphere to uncoated iron. In fact, the A.S.T.M. data show that the order of increasing corrosiveness for galvanized sheet is not the same as for black plate.

TABLE III.—COMPARISON OF OBSERVED AND CALCULATED TIME (IN YEARS) FOR APPEARANCE OF RUST.

Actual Average Weight of Coating, oz. per sq. ft.	Altoona		Brunot Island		Sandy Hook		State College	
	Observed	Calculated	Observed	Calculated	Observed	Calculated	Observed	Calculated
0.88	2.0	1.4	2.2	1.9	4.8	4.6	10.4	10.5
1.30	2.4	2.1	3.2	2.9	6.8	6.8	...	15.6
1.54	3.0	2.5	3.5	3.4	7.6	8.0	...	18.5
2.02	3.8	3.2	4.6	4.4	10.0	10.5	...	24.2
2.58	4.5	4.1	5.6	5.7	...	13.4	...	31.0

In discussing the usefulness of the several equations given, it should be pointed out that they are also subject to limitations and that unless these limitations are kept in mind false conclusions may be drawn. In the first place, the S-shape rate curve was obtained only at the four northern stations; the behavior of sheets exposed at Key West is quite different and is not described by Eq. 5. These relations do not therefore apply to all atmospheres. This is not a serious difficulty, however, because at least one rate curve is necessary to determine the index and such a curve itself indicates whether the equations are applicable. These equations are based on observations on hot-dipped sheets and are, of course, valid only for such material tested in the specified manner. Whether they will prove applicable to data on the rusting of other galvanized articles, and to modified test procedures, remains to be determined.

It must also be remembered that the equations represent data which, being the average of many measurements, should therefore be regarded as giving the life-expectancy for a group of galvanized sheets rather than as describing the appearance of rust on any individual sheet. In this respect they are precisely analogous to the life-expectancy data used by insurance companies. Finally, it should be pointed out that the method of treatment described is essentially empirical and that it can yield results no better than the observations to which it is applied. On this basis it simply demonstrates that the data collected by Subcommittee VIII can be generalized in a useful way. On the other hand, the mere fact that such a generalization is possible speaks well for the consistency of the observations and indicates that the committee has to a large extent overcome the considerable difficulties involved in making measurements of this sort.

SUMMARY

The data published by the Society on the average rate of development of rust on galvanized iron sheets exposed at Altoona, Pa., Brunot Island, (Pittsburgh), Pa., Sandy Hook, N. J., and State College, Pa., can be represented satisfactorily by the following equation:

$$\log (P/100 - P) = -(a + 12.3 \log w) + 10.7 \log t$$

where P is the percentage of the surface covered with rust after t years, w is the weight of the zinc coating in ounces per square foot, and a is an index which characterizes the corrosiveness of a given atmosphere for galvanized sheets as estimated by the A.S.T.M. test procedure. If the index a is known—and it can usually be determined from a sheet which rusts rapidly—the rate of appearance of rust in that atmosphere can be calculated for any weight of coating.

The rate curve for sheet with a given weight of coating exposed to a given atmosphere can be reduced to a straight line by the use of the autocatalytic scale as one coordinate and a logarithmic time scale as the other. A plot of this type facilitates interpolation, extrapolation and smoothing of the observations, as well as interpretation of the results.

DISCUSSION

Mr. W. Blum[1] (*presented in written form*).—The data treated in this paper represent the periods required for zinc to be dissolved from the steel, the corrosion of which usually occurs so rapidly that it may be used as an indication that the zinc has disappeared from the rusted areas. This behavior is observed only in those locations where the products of corrosion of the zinc are soluble, for example, in the sulfurous acid present in industrial atmospheres. In a purely marine location like Key West, Fla., insoluble zinc compounds such as basic zinc carbonate that are formed, retard the corrosion of the zinc, and may actually permit corrosion of the steel through pores in the zinc. This difference in behavior probably accounts for the fact that the equations derived by the author do not apply to Key West.

Equations 8 and 9 confirm previous observations that, in general, the life of a zinc coating in a given location is practically proportional to its thickness. These equations and conclusions apply equally to any given extent of corrosion, for example, to 1, 10, 50, or 90 per cent. Actually, little interest attaches to the rate of failure after 50 per cent or more has rusted.

If in a given location the resultant zinc compounds are soluble, if the zinc coating is uniform in thickness and composition, and if the conditions of exposure all over a sheet are uniform, the entire zinc coating would theoretically dissolve in the same period; that is, the curves in Fig. 1 would be vertical lines, and the position of each would represent the periods required for a zinc coating of that thickness to dissolve. The actual shape of the curves in Fig. 1 is the net result of (*a*) variations in local thickness of the zinc, (*b*) the presence of a zinc-iron alloy layer, and (*c*) variations in the environment on different parts of a sheet. An example of such a difference within a short distance is the fact that on small specimens, plated very uniformly with zinc, the zinc coatings almost invariably disappeared first (with resultant corrosion of the steel) along the lower edge, where the water deposited by rain or dew remained longest before it dried. As pointed out by the author, the shapes of the corrosion curves, and hence the numerical values of the constants, apply only to galvanized sheets prepared and exposed under specified conditions, and do not, therefore, have a wide or general usefulness.

Mr. C. D. Hocker[2] (*presented in written form*).—Mr. Austin develops a general equation which approximately defines the time required for any zinc-coated sheet to become covered with rust over any percentage of its area. The time, t, is expressed in terms of the weight of coating, w, and a constant, a, by the following equation:

$$\log (P/100 - P) = -a - 12.3 \log w + 10.7 \log t$$

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[1] Chemist, National Bureau of Standards, Washington, D. C.

[2] Plant Products Engineer, Bell Telephone Laboratories, Inc., New York, N. Y.

The constant, a, has a fixed value for any given atmosphere (4.1 for Altoona, 5.4 for Brunot Island, etc.).

Since this general equation has been found to work about equally well for defining the rusting performance of sheets in several atmospheres, it is considered probable that it will be found to work in other atmospheres for which data have not been collected. Presumably in determining the rust performance of sheets at a location hitherto untested, it would be necessary only to determine the value of a and then by substitution in the general equation, one could calculate the time required for a sheet carrying any weight of coating to become rust-covered over any percentage of its surface at the location involved.

In the main, this general equation works better for defining the times required to rust over 5 to 99 per cent of the area than it works for rusted areas of 1 to 5 per cent.

Observing that the equation does not work very satisfactorily at the 1 per cent rust points, Mr. Austin derives certain mathematical instrumentalities which will enable improved calculations for these 1 per cent points. If the general equation truly represents the rust performances, the times required for 1 per cent rusting would be

$$t_1 = a'w^{1.15} \text{ (Eq. 8 of paper)}$$

in which $a' = \text{antilog}\left(\frac{-2.00 + a}{10.7}\right)$.

Mr. Austin observes that Eq. 8 does not work as well as the following equation:

$$t_1 = a'w \quad \text{(Eq. 9 of paper)}$$

Equation 9 has no relation to Eq. 8, except that the exponent 1.15 is near to unity. Equation 9 is a simple statement of the relation, which has been expressed in A.S.T.M. reports, that the time for the beginnings of rust at any location is proportional to the weight of coating. Actually the times required for the A.S.T.M. sheets to rust to the extents of 2, 3, 5, 10, 25, and up to 50 per cent are also approximately proportioned to their weights of coating.

An investigator interested in the practical problem of predicting the times required for sheets at any untested location to become considerably rusted (up to at least 50 per cent) might proceed in either of two ways:

1. Making a few observations of the time required for light-coated sheets to rust to a per cent, P, and calculating that the times for heavier coated ones to rust to the same per cent, P, would be proportional to their coating weights.
2. Making a few observations on lightly coated sheets using the data to calculate the constant a. Use the value of constant a in calculating the time required for more heavily coated sheets to become rust covered over any proportion of their areas except the 1 per cent rust points. For these incident rust calculations, use the simplified formula $t_1 = a'w$, having calculated a' by the mathematical instrumentalities given by Mr. Austin.

To this writer it seems that the results should be about as good by the simpler method as by the more mathematical one.

Had Mr. Austin used a somewhat different method of approach, a general equation could be derived which would generally fit as well as the one he has formulated and which would work about as well at the 1 per cent points as at points further up on the curves. The derivation of Mr. Austin's general equation is based on the considerations which are outlined below:

The S-shaped curves depicting the original data must be symmetrical around the 50 per cent rust point in order to fit the basic equation

$$\log\left(\frac{P}{100 - P}\right) = k_1 + k_2 t$$

Most of them are not symmetrical and Mr. Austin chooses in effect to replot them on a P *versus* log t scale. While his paper does not show the replotted curves, it will be found that when such plots are prepared, most of the curves are made a little more nearly symmetrical. However, the replottings do not help greatly because in the range of values of t involved (about 2 to 10 yr.), the logarithm of numbers is too nearly proportional to the numbers themselves for the transformed curves to truly fit the basic equation

$$\log \frac{P}{100 - P} = k_1 + k_2 \log t.$$

When the P *versus* log t curves are transformed to the log $\frac{P}{100-P}$ *versus* log t graphs, straight lines similar to those of Figs. 2 and 3 result. Mr. Austin regards all of these straight lines to have slopes nearly enough alike so that he selects the figure 10.7 as the slope for all of them. Actually as the lines are drawn in Figs. 2 and 3, they have slopes ranging from about 8 to 13.5. There is opportunity for exercise of considerable individual judgment in the drawing of these straight lines. My own judgment of the fairest way to draw these lines does not make them parallel, but makes of each family a set of lines that are somewhat fan-shaped. My plottings result in lines that have slopes ranging from about 8 to 20, instead of all being close to 10.7.

The choice of a mathematical treatment which assigns the same slope to all of the log $\frac{P}{100-P}$ *versus* log t curves is a step which contributes much to the unsatisfactory fit of Mr. Austin's general equation at the 1 to 5 per cent rust points.

It might be expected that Mr. Austin's procedure of assigning a constant value, 10.7 to the k_2 constant would considerably invalidate the equating of the right- and left-hand terms in the basic equation: $\log \frac{P}{100-P} = k_1 + k_2 \log t.$ Actually the seriousness of the procedure is lessened because the errors tend to balance out. This is due to the fact that simple geometric considerations show that $k_1 = - k_2 \log t\ 50$ as well as being approximately equal to $(-2 - 12.3 \log w)$ as Mr. Austin deduces. As k_2 increases, k_1 increases, and since one is positive and the other negative, the total value of the terms on the right hand of the equation are not generally greatly affected by a deviation of k_2 from its true value.

If the derivation of a general equation is approached along the following lines, a better fitting relation is obtained:

1. Find a better way to transform the original data curves (curves like those of Fig. 1) into symmetrical curves than is afforded by changing these curves from P *versus* t curves to P *versus* log t curves. A better symmetry is arrived at by leaving the original curves untransformed but disregarding the data above 92 per cent rust points. Proceeding along the line, the general equation assumes the form $\log \frac{P}{92 - P} = k_1 + k_2 t.$

2. Do not attempt to make the log $\frac{P}{92-P}$ *versus* t curves fit a pattern of being parallel. Plot them as the data indicate and find ways of expressing both the k_1 and k_2 constants in terms of

w (weight of coating) and of numerical constants.

Following along the lines suggested above, one obtains the generalized equation shown below:

$$t = R(cw - 1.5) + 1.5$$

where:

R is a function of P, and is equal to $0.29 \log \frac{P}{92-P}$,

w = weight of coating, and

c is an atmospheric constant entirely independent of the mathematical derivation and is equal to the time required for a 1-oz. coated sheet to become rust covered over 20 per cent of its surface. The values of c are:
2.35 for Altoona
3.0 for Brunot Island, and
6.7 for Sandy Hook

The equation shown in the preceding paragraph holds about as well at 1 per cent rust points as it does at all points up to 91 per cent. It is not designed to hold above 91 per cent, and it appears rather unimportant from any practical standpoint to know how long it requires for any sheet to become rusted 95 or 100 per cent after it has already rusted to the extent of 91 per cent. This equation makes unnecessary the mathematical instrumentalities deduced by Mr. Austin to derive a constant a' from the constant a.

The method chosen by Mr. Austin to handle the American Society for Testing Materials sheet data as well as the one I have presented are merely two out of a variety of mathematical treatments that might be followed in deriving a generalized equation. An entirely different method of approach which will probably result in a suitably fitting generalized relation is the use of the equation

$$\log \tan P \text{ (or } 0.9P) = k_1 + k_2t$$

Any method of approach will result in at least one "atmospheric constant." The method of approach can be chosen so that the atmospheric constant is intimately tied up to the mathematical derivation (as is the case with Mr. Austin's constant a, which is equal to 10.7 multiplied by the logarithm of time for a 1-oz. sheet to become covered with rust over 50 per cent of its area); or a method of approach can be chosen which employs an atmospheric constant entirely independent of the mathematical derivation.

MR. J. B. AUSTIN[3] (*author's closure, by letter*).—Mr. Blum's very reasonable suggestion that the failure of Eq. 5 to represent the data for the station at Key West, Fla., is due to the fact that in a marine atmosphere the products of corrosion of zinc are insoluble basic compounds, is most interesting. On this basis, it is to be expected that Eq. 5 is chiefly applicable to atmospheres in which the products of corrosion are soluble.

Mr. Hocker is quite correct in stating that the method used in this paper is not the only way to approach the problem of generalizing the corrosion data and that alternative solutions might be equally satisfactory, or even better. Moreover, it is clear that the final choice of a method must inevitably involve some exercise of personal judgment, just as do the observations upon which the test results are based. The point to be emphasized, however, is that these results can be generalized in a useful way, a fact which does not seem to have received adequate recognition in the past.

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[3] Physical Chemist, Research Laboratory, United States Steel Corp. of Delaware, Kearny, N. J.

MEASUREMENT OF CORE LOSS AND A.C. PERMEABILITY WITH THE 25-CM. EPSTEIN FRAME

By S. L. Burgwin[1]

Synopsis

A combined core-loss and permeability test for small samples of Epstein strip 28 cm. long and about 1 lb. in weight has been developed and is now in use at the Westinghouse Research Laboratories. The test method is most useful for commercial frequencies and inductions above 10 kilogausses, but can be extended down to 1 kilogauss.

The method requires, in addition to the usual sine-wave voltage source and control, a special test frame with an air-flux compensating inductor, a reflecting type wattmeter, a synchronous rectifier, d-c. and a-c. milliammeters with calibrated resistors, and a mutual inductor for measuring peak exciting current. The construction and required characteristics of this apparatus is discussed in detail.

Two procedures for obtaining test data which have proven quite satisfactory are described step by step together with the necessary formulas for calculating the results.

Limitations of the apparatus and methods of minimizing their effects are discussed at some length.

Most acceptance data and many design data on the core loss and permeability of electrical sheet steel are obtained by the Epstein core-loss test, supplemented by either the Fahy or Burrows permeability tests, all of which have been adopted by the Society, and are described in the A.S.T.M. Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 – 39).[2]

In making acceptance tests where time and convenience of testing are important factors, the inconvenience of two separate test equipments and test specimens for core loss and permeability tests has been a necessary evil, largely because the uniformity of the sheet material as regards core losses has been such that a 5 or 10-kg. test sample was necessary to insure representative results. Speed in testing such large specimens necessitates the use of butt joints rather than alternate butt and lapped joints or double-lapped joints, and consequently prevents a combined core loss and permeability test, since permeability measurements are too greatly affected by the butt joints.

Improvement in uniformity and quality of magnetic characteristics of electrical sheet has made possible the use of much smaller test samples for obtaining representative results. Smaller samples and improved materials render the butt joint Epstein test less satisfactory, largely due to the effects of the joints. However, the same trend toward smaller samples and thus fewer strips, by decreasing the stacking time, makes it more feasible to use lapped joints which considerably minimize the effects of the joints and thus make a combined core-loss and permeability test possible.

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[1] Electrical Engineer, Westinghouse Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 516.

Growing interest in the testing of smaller samples suggests that a description and discussion of the test equipment and test methods evolved and used during the past 8 yr. at the Westinghouse Research Laboratories for testing small samples in the 25-cm. Epstein frame might be of value to those interested in the commercial testing of magnetic materials.

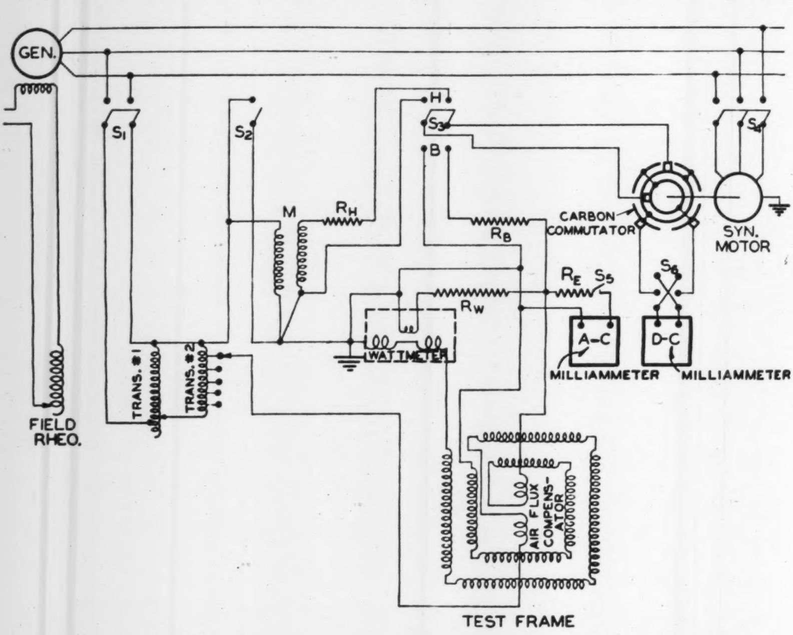


FIG. 1.—Circuit for 25-cm. Epstein Core-Loss and Permeability Tests.

TEST EQUIPMENT

Much of the equipment used in this test at the present time, while not standard, is, with the possible exception of the wattmeter, not difficult to construct. The circuit diagram of Fig. 1 shows the connections for all essential apparatus and will be followed in the ensuing discussion.

Power Supply:

The source of power is a three-phase, 120-v., sine-wave generator with 7.5 kva. capacity. This is the generator ordinarily used for the standard Epstein tests. Any low impedance source of power with a substantially sine-wave voltage would be equally satisfactory. In this case the generator is more convenient, as the field control provides an auxiliary voltage control.

Transformer No. 1:

The variable transformer is a 115-v., 2-kva. Variac. Optimum transformer capacity for the test is determined by the maximum impedance that can be tolerated in the primary circuit. In this case a 5-kva. Variac, which is now available would permit somewhat better wave form at high inductions.

Transformer No. 2:

A 5-kva. low impedance step-down transformer with 2-v. taps up to 20 v. on the low side would be ideal for this application. Actually a much larger auto transformer which happened to be available is being used with satisfactory results in the author's setup.

Test Frame:

In most of the core-loss testing at the Westinghouse Research Laboratories, a test frame with a 352-turn primary and secondary has been used. The structural details of such a frame may be observed in Fig. 2. In order that the reader will not be misled by the six binding posts, it might be mentioned that this particular frame has an additional winding to be used as a shield in high frequency tests or as a d-c. winding in superposed a-c. and d-c. tests. This additional winding is, of course, unnecessary for the 60 cycle core loss and permeability test.

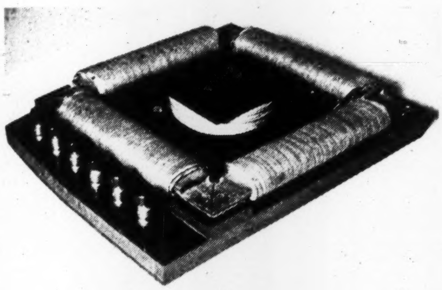


FIG. 2.—25-cm. Epstein Frame Used in Westinghouse Research Laboratories.

The base of the frame consists of a 12 by 14 by $\frac{1}{2}$-in. Micarta board. The corners of the test coils are supported by 2 by 2 by $\frac{1}{2}$-in. Micarta blocks. The coil forms are rectangular Micarta tubing $1\frac{1}{4}$ by $\frac{1}{4}$-in. inside dimensions with a $\frac{1}{16}$-in. wall. The length of each tube is such that, when cut at a 45-deg. angle at both ends, the mean length is 25 cm. The top and sides of each end are cut back so that the enclosed portion of the tube is 21 cm. long. Available winding length is 20 cm.

In the frames used by the author, primary and secondary windings consisted of 88 turns per leg of No. 13 single cotton-covered enameled wire. This wire, closely wound, results in a coil length per leg of 19 cm. However, there are several advantages which will appear later in using windings of 175 instead of 88 turns per leg. For such windings the author recommends No. 19 single cotton-covered enameled wire for the secondary, to be wound next to the Micarta tube, and a primary of four layers of the same size wire wound on top of the secondary. Connections between legs should be transposed at each corner and the four layers connected in parallel. Coil turns with these wire sizes will have to be kept as close as possible to keep within the available 20-cm. winding space. In taking out leads from these coils, the best practice is to start both primary and secondary windings at the same corner as shown in Fig. 1. This practice makes it possible to keep the voltage between primary and secondary windings very low, and while not strictly necessary at 60 cycles, may save trouble if the frame should be used at higher frequencies.

The greatest source of trouble experienced in building some six or seven test frames has been, strangely enough, errors in counting the exact number of turns. Probably counting turns appears to be so simple that insufficient care is taken.

Air-Flux Compensating Inductor:

The small air-core inductor mounted in the center of the test square (Fig. 2) is connected as shown in Fig. 1. Its purpose is to balance the mutual inductance between the primary and secondary of the test frame without iron, and thus compensate for air flux in the measurement of secondary voltage.

The construction and mounting of the air-flux compensating inductor is shown in Fig. 3. Approximately a one to five ratio of primary to secondary turns is used and the primary is wound inside to keep its resistance and self inductance low.

For the 352-turn frame, two layers of eleven turns each of No. 13 single cotton-covered enameled wire wound upon a 2-in. diameter Micarta tube comprise the primary winding. The secondary is wound on top of the primary and consists of ten layers or approximately 110 turns of the same size wire. The number of secondary turns is varied until the mutual inductance just balances that of the test frame without iron. The total cross-section of such a primary and secondary winding is approximately 1 in. square. The mutual inductance of this inductor is approximately 100 microhenries, and the self-inductance of the primary approximately 20 microhenries.

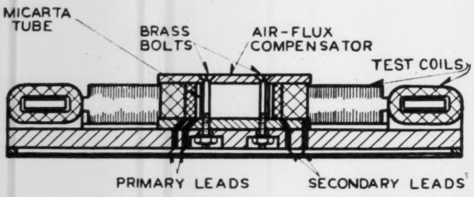


FIG. 3.—Cross-Sectional View of Test Frame to Show Mounting of Air-Flux Compensator.

For the 700-turn frame the physical dimensions of the compensator remain the same, but the primary is increased to 4 layers or 44 turns of No. 13 wire and the secondary to 10 layers or 220 turns of No. 19 single cotton covered enameled wire. This results in a mutual inductance of approximately 400 microhenries and a primary self-inductance of 80 microhenries.

In connecting the leads from the mutual inductor to those of the test frame, it is best practice—but not strictly necessary—to connect the finish of the primary winding of the test frame to the finish of the primary of the inductor winding and the finish of the secondary test winding to the start of the secondary inductor winding (see Fig. 1) as then the adjacent layers of the primary and secondary inductor windings can be kept at approximately the same potential.

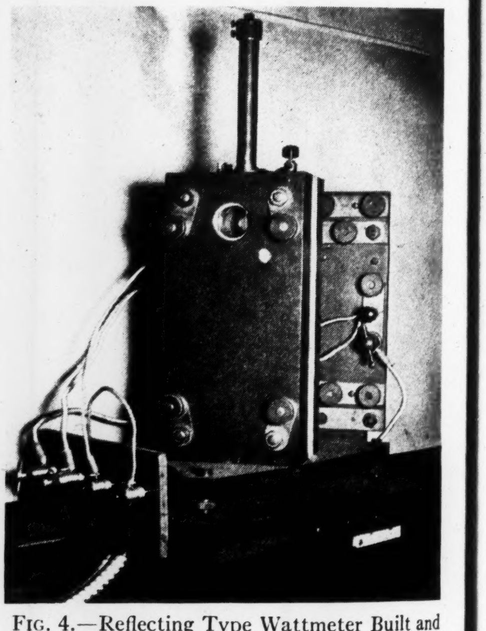


FIG. 4.—Reflecting Type Wattmeter Built and Used at the Westinghouse Research Laboratories.

Wattmeter:

The wattmeter used at the Westinghouse Laboratories for these tests is a light-beam reflecting dynamometer built in the laboratories (Fig. 4). The author has been informed that reflecting type wattmeters can be obtained from at least two companies and that another is contemplating placing such a wattmeter on the market.

The desirable limiting values for essential constants of the wattmeter for the 352 and 700-turn test frames are given in Table I.

These specifications are in excess of requirements for satisfactory core-loss testing, which may be objectionable since too high sensitivity often tends toward decreased ruggedness and stability. However, experience with the wattmeter of Fig. 4 which is quite satisfactory from the standpoint of ruggedness and stability indicates that these specifications can be met satisfactorily. The wattmeter of Fig. 4 satisfied the specifications for the 352-turn frame, except that its sensitivity is one-half of that specified. However, the current capacity is 50 amp., so that if the turns of the current coils were doubled, this wattmeter would completely satisfy the specifications. Wattmeters satisfying the above specifications could be used satisfactorily over a range of induction from 1 to 17 kilogausses with a 1-lb. specimen.

TABLE I.

Characteristics	352-turn Test Frames	700-turn Test Frames
Minimum sensitivity with 1000 ohms in potential circuit, watts per radian	4	1
Maximum potential coil resistance, ohms	125	500
Maximum potential coil inductance, millihenries	0.5	2
Current capacity, amp.	20	10
Maximum current coil inductance, microhenries	10	50
Maximum phase angle between current and effective field at 60 cycles, radians	1×10^{-4}	1×10^{-4}

Mutual Inductor:

The mutual inductor, M, in the circuit of Fig. 1 is used with an average voltmeter across its secondary to measure peak exciting current which, as will be explained later, is regarded as proportional to magnetizing force H.

Details of construction of an inductor built by the author for use with the 352-turn frame are shown in Fig. 5. Dimensions are such that the cross-section of primary and secondary windings is approximately 2 in. square, which makes the over-all diameter approximately 8 in. These seemingly excessive dimensions are due mainly to the 30-amp. current capacity for which the inductor is designed and would have been much greater had not the ratio of primary to secondary turns been kept as low as possible.

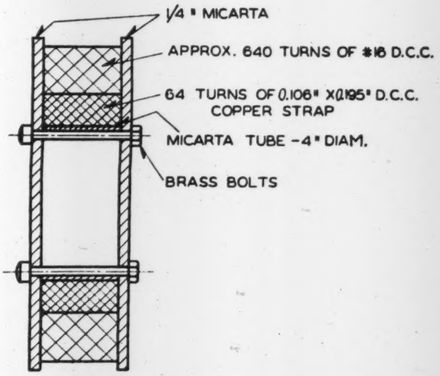

1/4" MICARTA
APPROX. 640 TURNS OF #16 D.C.C.
64 TURNS OF 0.106" X 0.195" D.C.C.
COPPER STRAP
MICARTA TUBE -4" DIAM.
BRASS BOLTS

FIG. 5.—Construction of Mutual Inductor.

Constants for this inductor are:

L_1 = 0.495 millihenry L_2 = 78 millihenries
R_1 = 0.050 ohm R_2 = 5.9 ohms
M = 3.67 millihenries

The value of mutual inductance was not particularly well chosen, as a value of 4.19 millihenries would have made the calculation of the value of R_H (see Fig. 1) somewhat simpler as will be shown later. Except for this slight inconvenience, the inductor proved quite satisfactory for use in the circuit of Fig. 1.

An inductor for use with a 700-turn test frame should have somewhat different constants. A convenient value of mutual inductance would be 8.33 millihenries. A 10-amp. current capacity should be satisfactory, and a one-to-five ratio of primary to secondary turns would keep the size within reasonable limits for such a current capacity without increasing the secondary self-inductance enough to seriously affect the accuracy of measurement of peak current. Another reason for the selection of such a ratio is that the self-inductance of the primary is thus kept low enough to permit switching the inductor into the test circuit without greatly affecting the setting of induction in the test specimen.

A design embodying these features having the same general construction as the inductor of Fig. 5 requires a primary winding of 8 layers of 19 turns per layer of No. 12 single cotton-covered enameled wire, wound upon a 3-in. diameter tube 1.75 in. long, and a secondary winding of 800 turns of No. 20 single cotton-covered enameled wire wound in 19 layers of 44 turns per layer over the primary winding. The self-inductance of the primary, as calculated, should be 1.85 millihenries, only four times that of the test frame, and of the secondary 84.5 millihenries which, as will be shown later, is not sufficient to cause a serious error in the measurement of peak current. The calculated resistance of the primary is 0.24 ohm and of the secondary 11.2 ohms.

Some improvements could be made in this inductor design by using a core of more solid construction and by using a secondary of wire with a low temperature coefficient of resistance. At present the effect of any variation of the copper resistance is swamped out by an external series resistance of at least 1000 ohms. It would be possible if the secondary winding had a negligible temperature coefficient to decrease the wire size used until the secondary resistance was a large part of the 1000 ohms. This should not only decrease the physical size of the inductor, but should also reduce the secondary self-inductance by improved coupling. These refinements were not incorporated in the author's design, as other factors limit the precision of measurement of peak current for high induction permeability measurements to such an extent that, in the author's opinion, the additional trouble and expense are not justified.

Fluxmeter:

A fluxmeter or average voltmeter for measuring the average voltage of the secondary of the test frame and of the mutual inductor consists in general of a rectifier and d-c. voltmeter. Copper-oxide rectifiers, vacuum tubes, and even crystals have been used, but in the author's opinion an arrangement consisting of a synchronous reversing switch d-c. milliammeter, and external resistor connected as shown in Fig. 1, is more reliable, more flexible, and requires less attention. The d-c. milliammeter used by the author has a 0 to 1 milliampere range, although a 0 to 2 range would be more convenient for direct calibration in kilogausses. This meter must have no shunt, because the current through the meter is not steady, but oscillating. Under such conditions, a meter having a resistance shunt will not in general give the correct reading.

The synchronous reversing switch consists of a carbon commutator rotated by a synchronous motor. The carbon brushes used with the commutator are fixed relative to each other, so that the circuit through the milliammeter is reversed every 180 electrical degrees, but can be moved about the commutator so that reversal occurs at any desired phase position. Carbon-to-carbon contacts are used because they have much lower and more constant contact resistances for very low currents than carbon-to-metal or metal-to-metal contacts.

The commutator built by the author has four segments and is driven at 1800 rpm. It consists of two carbon disks approximately 6 in. in diameter and 1 in. thick, both of which have one face plain and two opposite quadrants milled out of the other face to the depth of $\frac{1}{2}$ in. The two milled faces are fitted together with mica insulation between and the disks clamped between two steel plates from which they are insulated by mica washers. This results in a carbon commutator $\frac{1}{2}$ in. in width between two $\frac{1}{2}$-in. carbon collector rings.

The main essentials in mounting the two commutator brushes are that they maintain a relative angular distance of 90 mechanical degrees or 180 electrical degrees to each other, and that they can be moved together easily, preferably by a worm gear, to any phase position. Brushes for the commutator just described can be as much as $\frac{1}{8}$ in. in thickness without causing appreciable error in measuring the average of a sine or peaked voltage wave. The brushes should have small enough inertia to follow radial variations in the commutator without requiring excessive pressure.

Root-Mean-Square Voltmeter:

The root-mean-square voltmeter shown in Fig. 1 consists of a milliammeter and external resistor, and is used in the measurement of the form factor of the secondary voltage. Switch S_5 cuts out the voltmeter when wattmeter readings are made to prevent excessive instrument losses, since a-c. voltmeters require much more electrical energy for operation than corresponding d-c. voltmeters.

Measurement of root-mean-square voltage does not have to be nearly so precise as the measurement of average voltage, since core-loss values for most magnetic materials are relatively insensitive to small changes in form factor as long as the average voltage is constant. However, it may be desirable to use an accurate root-mean-square voltmeter of the dynamometer type, which has a stable calibration, in order to check the average voltmeter, if the average voltmeter is of the type utilizing a copper-oxide or vacuum-tube rectifier. With the carbon commutator circuit of Fig. 1, the author has found a thermal voltmeter, or more precisely, a thermal milliammeter (10 ma. full scale, with an external variable resistor) as is shown in Fig. 1, to be satisfactory from the standpoint of accuracy, although it is doubtful that such an instrument is sufficiently rugged for commercial applications.

With a 700-turn frame 0 to 15 and 0 to 30 voltage ranges are necessary. For a 352-turn frame these ranges are, of course, halved.

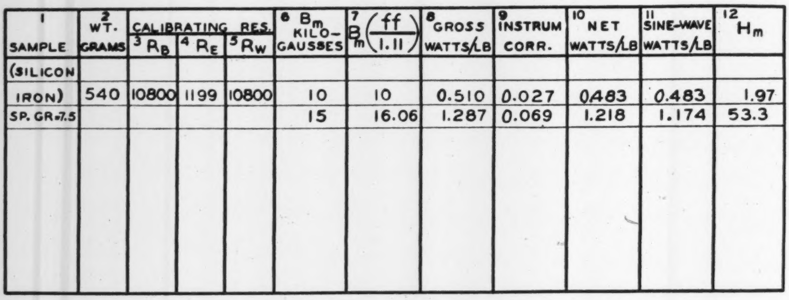
Resistors:

In Fig. 1, four resistors, R_H, R_B, R_E, and R_W are shown. These resistors for maximum convenience should be of the five dial decade type with a 100,000-ohms maximum resistance.

TEST PROCEDURE

Although it is possible to test various lengths and widths of strip material in the 25-cm. Epstein frame, the Epstein width of 3 cm. and a length of 28 cm. has been used by the author except for special research problems. Others prefer somewhat longer strips because the greater length facilitates stacking and removing the strips. However, it has seemed best to design the test method and apparatus for testing the 28-cm. length which is, of course, the minimum length for normal double lapped joint tests in a 25-cm. test frame. This does not, of course, preclude testing of longer strips, but simply makes 28 cm. the most convenient test length for this test design.

The test frame described earlier accommodates a 24-strip specimen of 14-mil material stacked with double-lapped joints. The weight of such a sample of silicon iron is approximately 1.2 lb., or 540 g. To illustrate the test procedure, the steps in testing such a 540-g. specimen of silicon iron will be described in detail.



1 SAMPLE	2 WT. GRAMS	CALIBRATING RES.			6 B_m KILO-GAUSSES	7 $B_m\left(\frac{ff}{1.11}\right)$	8 GROSS WATTS/LB	9 INSTRUM CORR.	10 NET WATTS/LB	11 SINE-WAVE WATTS/LB	12 H_m
		3 R_B	4 R_E	5 R_W							
(SILICON											
IRON)	540	10800	1199	10800	10	10	0.510	0.027	0.483	0.483	1.97
SP. GR=7.5					15	16.06	1.287	0.069	1.218	1.174	53.3

FIG. 6.—Typical Data Sheet for First Method.

The specimen is weighed and then stacked in the test frame in a manner similar to that described in the A.S.T.M. Standards for the low-induction a-c. tests.[2] The resulting joints have been called double-lapped joints by the author, due to the double thickness of material in each joint and have been discussed elsewhere.[3]

Two methods of testing may now be followed with the circuit of Fig. 1. In the first, values of R_B, R_E, and R_W are calculated for each specimen such that the corresponding meters are made to read directly in kilogausses, kilogausses multiplied by the ratio of the actual form factor to 1.11, and watts per pound, respectively. In the second these resistors are set at values such that the meters read average volts, root-mean-square. volts, and watts, respectively, and values of these quantities are calculated or measured for each specimen. Each method has advantages for particular applications so that both will be described in detail.

First Method:

A typical data sheet for the first method is shown in Fig. 6. The test procedure will be described with this sheet as a guide.

The calibrating resistances, columns 3, 4, and 5, are calculated from the following formulas:

$$R_B = \left(\frac{4 f N_2 \times 10^{-8}}{4 l \times (\text{sp. gr.})}\right)\left(\frac{B_m}{I_{d.c.}}\right)(\text{wt.}) \quad (3)$$

where

f = frequency,
N_2 = secondary turns of the test frame,
wt. = weight of sample from column 2,
l = average length in centimeters of the strips in the sample.
sp. gr. = specific gravity of sample (See A.S.T.M. Standards),[2] and
$I_{d.c.}$ = the rectified current flowing through the d-c. milliammeter (average value).

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[3] S. L. Burgwin, "A Method of Magnetic Testing for Sheet Material," *Review of Scientific Instruments*, Vol. 7, July, 1936, p. 272.

$$R_E = \left(\frac{4.44\, f\, N_2 \times 10^{-8}}{4 \times l \times (\text{sp. gr}).}\right)\left(\frac{B_m}{I_{r.m.s.}}\right)(\text{wt.}) \quad (4)$$

where

$I_{\text{r.m.s.}}$ = the root-mean-square current flowing through the a-c. milliammeter, and

B_m = test inductions in kilogausses.

$$R_W = (K_1)(K_2)\,\frac{\text{wt.} \times l_m}{454 \times 4 \times l} \ldots\ldots (5)$$

where

K_1 = the resistance of the wattmeter potential circuit required to give a sensitivity of one watt per scale division,

K_2 = desired wattmeter sensitivity in watts per pound per scale division, and

l_m = length of magnetic path (assumed to be 94 cm.)

Eq. 5 simplified becomes

$$R_W = \left(0.0518\,\frac{K_1}{l}\right)(K_2) \times (\text{wt.}) \ldots (5a)$$

It should be noted that in the right-hand member of each of the above equations the first quantity in parenthesis will be a constant for a series of tests upon similar samples at a given frequency, the second quantity is arbitrarily selected for convenience and satisfactory sensitivity, and the third quantity alone varies from sample to sample. Thus for 60-cycle tests with a 700-turn test frame upon a 28-cm. sample with a calibration of 10,000 and 1000 gausses per milliampere for the d-c. and root-mean-square milliammeters, respectively, Eqs. 3 and 4 reduce to

$$R_B = 20 \text{ wt.} \ldots\ldots\ldots\ldots (3a)$$

$$R_E = 2.22 \text{ wt.} \ldots\ldots\ldots\ldots (4a)$$

Similarly, if the wattmeter has a sensitivity of 1 watt per radian with 1000-ohms resistance in the potential circuit, a calibration of 10.82 watts per pound per radian reduces Eq. 5*a* to

$$R_W = 20 \text{ wt.} \ldots\ldots\ldots\ldots (5b)$$

A millimeter scale placed at a distance in this case of 108.2 cm. from the wattmeter will then have a calibration of 0.01 watts per lb. per mm., which is a convenient value for many tests. Under the conditions specified, $R_B = R_W = 10{,}800$ and $R_E = 1199$ for a 540-g. sample.

Values in column 6 (Fig. 6) represent arbitrary test inductions.

Up to this point all values appearing in the data sheet have been calculated or arbitrarily chosen. Values in columns 7, 8, and 12 are to be obtained from the test. Proceeding to the testing, it is assumed that the sample has been properly stacked in the test frame, after which resistors R_B, R_E, and R_W (Fig. 1) are set at the calculated values minus in each case the internal resistance of the corresponding meter; switches S_1, S_2, S_4, and S_5 are closed and switch S_3 is placed in position *B*. The generator field rheostat and transformers Nos. 1 and 2 are adjusted until the d-c. milliammeter indicates that the desired induction, column 6, has been obtained. (When using a synchronous commutator, care must always be taken that the brushes are in the proper position to give a maximum reading of the d-c. milliammeter). Under these conditions, values in column 7 are taken from the reading of the a-c. milliammeter. Typical values are shown.

To obtain the wattmeter readings recorded in column 8, switch S_5 is opened and the generator field rheostat or transformer No. 1 readjusted if necessary to maintain the correct reading of the d-c. milliammeter. The purpose of opening switch S_5 is to reduce instrument burden and consequently the necessary correction.

Since column 8 is in watts per pound, the instrument correction will also have to be in watts per pound. That is, the total copper loss of the secondary circuit which, in this case, will be almost entirely in the wattmeter potential circuit and in the d-c. milliammeter circuit, must be divided by the active weight of the sample, as follows:

$$\text{Instrument losses per pound} = \left(\frac{4 \times (\text{Form Factor})\, f\, B_m\, N_2\, \text{wt.}}{(\text{sp. gr.})\, l \times 4 \times 10^8}\right)^2 \left(\frac{454 \times 4 \times l}{\text{wt.} \times l_m}\right)\cdot\left(\frac{1}{R_B} + \frac{1}{R_W}\right) \ldots (8)$$

It will be noted that since R_B and R_W are proportional to the weight of the sample, the instrument losses per pound do not depend upon the weight of the sample. That is, if the equations for R_B and R_W are written

$$R_B = K_3\,(\text{wt.}) \ldots\ldots\ldots\ldots (3b)$$

and

$$R_W = K_4\,(\text{wt.}) \ldots\ldots\ldots\ldots (5c)$$

$$\text{Instrument loss per pound} = \frac{227 \times 10^{-16}\, \pi^2 f^2\, N_2^2}{(\text{sp. gr.})^2\, l\, l_m} \times \left(\frac{1}{K_3} + \frac{1}{K_4}\right)\left(\frac{FFB_m}{1.11}\right)^2 \ldots\ldots (8a)$$

where FF = form factor.
For the example under consideration, substituting values from Eqs. *3a* and *5b* in Eq. *8a*,

$$\text{Instrument loss per pound} = 2.67 \times 10^{-9}\left(\frac{1}{20} + \frac{1}{20}\right)\left(\frac{FFB_m}{1.11}\right)^2$$

$$= 2.67 \times 10^{-10}\left(\frac{FFB_m}{1.11}\right)^2 \ldots\ldots\ldots\ldots (8b)$$

Thus the instrument losses for a given induction are obtained by multiplying the square of the values of column 7 by a constant factor.

The values of column 10 are obtained by subtracting column 9 from column 8.

At the higher inductions when the form factor of the voltage wave is greater than 1.11, a correction in the values of net watts per pound to a sine-wave basis is desirable. The method used has been described by Camilli,[4] but since the equation used here is of slightly different form, its derivation will be given.

If it can be assumed that the equation

$$P_1 = K_5 + K_6(FF_1)^2 \ldots\ldots\ldots (10)$$

represents the relation of the measured core loss P_1 to the measured form factor FF_1 then

$$P_0 = K_5 + K_6(1.11)^2 \ldots\ldots\ldots (10a)$$

is the value of core loss for a sine wave of induction. Furthermore, if for a given material the equation

$$\frac{K_6(1.11)^2}{P_0} = K_7 \ldots\ldots\ldots (10b)$$

can be assumed to hold, that is, if K_5, K_6, and K_7 are constants, it is possible, knowing K_7, P_1, and FF_1, to calculate P_0 from the equation:

$$P_0 = \frac{P_1}{1 + K_7\left[\left(\frac{FF_1}{1.11}\right)^2 - 1\right]} \ldots (10c)$$

This equation can be simplified with negligible error when the form factor differs from 1.11 by less than 15 per cent to

$$P_0 = P_1\left[1 - 2\,K_7\left(\frac{FF}{1.11} - 1\right)\right] \ldots (10d)$$

The constant K_7 in these equations is usually thought of as the fraction of the losses due to eddy currents. This conception cannot be strictly justified, and may lead to errors, so that it is best to evaluate constant K_7 by measuring two values of core losses at the same induction but with different wave forms. This is easily accomplished practically by measuring the core losses first with switch S_2, Fig. 1, closed and then with it open. The value of the constant may be calculated from

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[4] G. Camilli, "A Flux Voltmeter for Magnetic Tests," *Transactions*, Am. Inst. Electrical Engrs., Vol. XLV, January, 1926, pp. 721–728.

$$K_7 = \frac{P_2 - P_1}{P_1\left[\left(\frac{FF_2}{1.11}\right)^2 - 1\right] - P_2\left[\left(\frac{FF_1}{1.11}\right)^2 - 1\right]} \qquad (10e)$$

It should also be noted that in all of these equations the ratios of the measured form factors to 1.11 are the ratios of the values of column 7 to the corresponding values of column 6, Fig. 6.

For the example under consideration, no correction need be made at 10 kilogausses, but at 15 kilogausses, assuming $K_7 = 0.25$,

$$P_0 = 1.218\left[1 - 2 \times 0.25\left(\frac{16.06}{15} - 1\right)\right]$$

$$= 1.174 \text{ watts per pound}$$

This completes the core-loss test by the first method.

In column 12 of Fig. 6, values of maximum magnetizing force H_m are recorded. It is assumed that H_m is proportional to the maximum exciting current I_m. This quantity may be measured[5] after the core-loss test by opening switch S_2, Fig. 1, readjusting the field rheostat or transformer 1, if necessary, to maintain the correct reading on the d-c. milliammeter, and then switching S_3 to position H.[6] The reading of the d-c. milliammeter will then be proportional to H_m if R_H is large compared to the reactance of the secondary winding of M.

For the sake of convenience, a value of R_H may be selected such that the magnetizing force is related to the d-c. milliammeter reading by some multiple of ten. With this in mind a formula for R_H whose derivation will be discussed in Appendix I[7] is:

$$R_H = \frac{10fMl_m}{\pi N_1}\,\frac{H_m}{I_{\text{d.c.}}} \quad \ldots\ldots\ldots (12)$$

where

M = mutual inductance of inductor M, Fig. 1,

l_m = assumed length of magnetic path (for inductions above 12 kilogausses, 88 cm. is assumed instead of 94 cm., as used in the core-loss test),

N_1 = primary turns of the test frame, and

$I_{\text{d.c.}}$ = average current in d-c. milliammeter.

When M = 8.33 millihenries, f = 60 cycles, l_m = 88 cm., and N_1 = 700 turns, this formula reduces to

$$R_H = 0.2\,\frac{H_m}{I_{\text{d.c.}}}$$

Thus, if it is desired to make one milliampere represent 100 oersteds,

$$\frac{H_m}{I_{\text{d.c.}}} = \frac{100}{0.001} = 100{,}000$$

and

$$R_H = 20{,}000 \text{ ohms}$$

Typical test values appear in column 12, Fig. 6.

Second Method:

The alternative method of test procedure is much like the first, except that the values of R_B, R_E, and R_W are such that the d-c. milliammeter reads directly in average volts, the a-c. milliammeter in root-mean-square volts, and the wattmeter in watts. Consequently, average volts, watts per pound, and instrument losses must be calculated separately for each test sample, instead of values of R_B, R_E, and R_W as in the first method.

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[5] G. Camilli, "Reduction of Transformer Existing Current to Sine Wave Basis," *Transactions*, Am. Inst. Electrical Engrs., Vol. LXVI, May, 1927, pp. 692-696.

[6] When using a synchronous commutator, a sharper adjustment can be obtained by adjusting the position of the brushes for a maximum reading of the d-c. milliammeter with switch S_3 on H rather than on B, because the wave shape of H is peaked, while that of B is flat-topped.

[7] See p. 794.

A sample data sheet is shown in Fig. 7 and again the test procedure will be described with the test sheet as a guide.

Under column 15 is the active weight of the sample in pounds, which is obtained from column 2 by the equation

$$\text{Active weight} = \frac{l_m}{454 \times 4 \times l} \text{ (total weight)} \ldots (13)$$

where

l_m = length of magnetic path (94 cm.), and

l = average length of strips in the sample.

When $l_m = 94$ cm. and $l = 28$ cm., this equation becomes

$$\text{Active weight} = 1.85 \times 10^{-3} \text{ (total weight)} \ldots (13a)$$

The arbitrary test inductions are placed under column 16.

Values of average voltage, column 17, are calculated for each sample and each test induction from the equation

$$E_{\text{avg.}} = \frac{4 f B_m N_2 \text{ wt.}}{10^8 \times 4 \times l \times \text{(sp. gr.)}} \ldots (14)$$

when $f = 60$ cycles, $B_m = 10{,}000$ gausses, $N_2 = 700$ turns, $l = 28$ cm., and sp. gr. = 7.5, this equation reduces to

$$E_{\text{avg.}} = 0.02 \text{ (wt.)} \ldots\ldots\ldots (14a)$$

Values of $E_{\text{r.m.s.}}$, column 18, are test values obtained from the a-c. milliammeter, or in this case voltmeter, when the d-c. milliammeter, or voltmeter, reads the value calculated from Eq. 14.

Values of gross watts, column 19, are also test values obtained with switch S_5 open as in the previous method, the difference being that now the wattmeter is calibrated in watts instead of watts per pound.

13	14	15	16	17	18	19	20	21	22	23	24
SAMPLE IDENT.	WEIGHT		B	E AVG.	$E_{\text{R.M.S.}}$	GROSS WATTS	INST. LOSS	NET WATTS	NET WATTS / LB.	SINE-WAVE WATTS/LB	H_m
	TOTAL G.	ACTIVE LB.									
SILICON	540	1.00	10	10.80	12.00	0.512	.0288	0.483	0.483	0.483	1.97
IRON			15	16.20	19.23	1.292	.074	1.218	1.218	1.174	53.3
(SP. GR =											
7.5)											

FIG. 7.—Typical Data Sheet for Second Method.

The instrument losses, column 20, are in watts and are calculated from the measured root-mean-square voltage by means of equation

$$\text{Instrument losses} = E^2_{\text{r.m.s.}} \left(\frac{1}{R_B} + \frac{1}{R_W} \right) \ldots (15)$$

The net watts, column 21, are obtained by subtracting the values of column 20 from the corresponding values of column 19.

Net watts per pound, column 22, is the quotient of net watts, column 21, divided by the active weight, column 15.

Net watts per pound must be corrected to a sine-wave basis, as explained in the description of the first method. Equations 10*c* or 10*d* may be used without change. The form factor, FF_1, in these equations will, of course, be the ratio $E_{r.m.s.}/E_{avg.}$ The corrected values are recorded in column 23.

The values of H_m, column 24, are obtained as in the first method.

In the second test method, supposing the d-c. milliammeter to be 2 ma. full scale, the root-mean-square milliammeter 20 ma. full scale, and the wattmeter to have the same sensitivity, as assumed for the first method, convenient values of R_B, R_E, and R_W would be

$$R_B = 10{,}000 \text{ ohms}, R_E = 1000 \text{ ohms, and } R_W = 10{,}000$$

provided the wattmeter scale were at a distance of 1 meter. The typical values recorded in the data sheet, Fig. 7, are calculated for these conditions.

DISCUSSION

Uniformity of Magnetization:

The test method just described involves a number of assumptions which need to be examined. First, the uniformity of magnetization of the specimen will be discussed. Two factors which influence this uniformity are the reluctance of the joints and the uniformity of distribution of the magnetizing ampere turns. The reluctance of double-lapped joints, although not negligible, is generally less than that of an equal length of the sample between joints for the usual commercial materials, so that in this regard the double-lapped core is better, even at low inductions, than a square without joints. Lack of uniform distribution of ampere turns is more serious. Due to structural reasons the magnetizing winding can cover only about 80 per cent of the magnetic path. This means that a certain amount of leakage flux cannot be avoided. The nonuniformity in magnetization caused by this leakage flux will be roughly in an inverse ratio to the permeability and to the number of test strips of the sample. Since the number of strips in a double-lapped joint test is limited practically by stacking considerations, an appreciable nonuniformity in magnetization can be expected at high inductions, where the permeability is extremely low. Measurements of the uniformity of magnetization in the 24-strip double-lapped joint test as compared with the standard Epstein test upon a 5-kg. specimen and the standard Fahy permeammeter test indicate a more uniform magnetization than in either of these standard methods. Therefore, less error should be expected from nonuniformity in the legs between joints in the double-lapped joint core-loss and permeability tests than in the standard Epstein and Fahy permeability tests.

Joint Losses:

In the core-loss test of the double-lapped joint core, losses in the corners cause some difficulties. If these joint losses are completely neglected and the magnetic path considered to be 88 cm., the measured core losses per unit weight of sample may ordinarily be expected to be from 4 to 10 per cent[3] high, depending upon the permeability of the sample. For this reason a 7 per cent correction is arbitrarily made and the magnetic path assumed to be 94 cm. Results based upon this assumption happen to agree reasonably well with the result of the 5-kg. Epstein test for commercial material, so that substitution of the 25-cm. Epstein test for the standard 5-kg. test does not necessarily invalidate experience factors based upon the older test method.

Air Compensator:

In the preceding text it has been assumed that the quantity measured by the average voltmeter is normal induction; actually it is intrinsic induction, since the air-flux compensator compensates for all the air flux within the test winding with no sample present. The error in the measurement of core losses due to this assumption is generally negligible up to 16 kilogausses. At higher inductions corrections must be made. It should also be mentioned at this point that balancing the mutual inductance of the compensator against that of the test frame does not give quite accurate compensation, although there is some evidence that the error resulting from this method is considerably less than from calculating the compensating inductance from the physical area of the test windings.

If corrections are necessary, normal induction can easily be obtained by adding the test value of H_m to the measured value of induction. This may be desirable, particularly in permeability testing at high inductions, as the error in permeability may be appreciable.

Wave Form of Induction:

The correction of core losses to a sine-wave basis is really an extrapolation of a curve of losses *versus* the square of the form factor. This curve is supposed to be linear, but actually is not for many materials. Furthermore, distortion of the wave-form by adding resistance to the primary circuit gives a slightly different curve than the addition of reactance; consequently to avoid excessive extrapolation it is best to keep the deviation from a sine wave of induction as small as possible. The impedance of the primary circuit, and particularly the reactance, determines the distortion of the induction wave in the test sample. In a 24-strip test specimen the cross-sectional area of the test specimen is about one tenth the cross-sectional area of the magnetizing winding, so that approximately nine-tenths of the self-inductance of the primary winding is effective in distorting the induction wave. Therefore, in designing this test circuit there is not much point in keeping the effective inductance of the circuit in series with the test frame below one fifth of the inductance of the test frame. In the case of the 700-turn test frame, this external inductance should, therefore, not be more than 0.1 millihenry. Reactance in this range appears to be about ten times as effective as resistance in distorting the flux wave, so that the effective resistance external to the test frame should not be greater than 0.5 ohm.

Measurement of Permeability:

For a-c. applications it is desirable to design and to rate magnetic materials on a basis of apparent watts per pound rather than permeability. In the test method described in the preceding text, this quantity can be readily calculated for inductions up to the knee of the *B-H* curve, which, for most commercial materials, occurs at about 12 kilogausses, from measurements of root-mean-square exciting current, utilizing the equation

$$\text{Apparent watts per pound} = E_{\text{r.m.s.}} I_{\text{r.m.s.}} \frac{454 \times 4 \times l}{\text{wt.} \times l_m} \quad (16)$$

where

l = average length of the strips (28 cm.),

l_m = assumed length of magnetic path (94 cm.), and

wt. = total weight in grams.

It is preferable to use a thermal type ammeter for measuring the root-mean-square exciting current, as impedance of thermal meters, besides being less than for other types, is almost wholly resistance, which affects the value of root-mean-square exciting current at a given induction less than reactance.

At higher inductions, the distortion of the induction wave due to the impedance of the ammeter and the rest of the primary circuit may be sufficient to increase the root-mean-square exciting current for a given maximum induction by as much as 50 per cent over the value for sine-wave variation of induction. Thus ordinarily for inductions above 12 kilogausses it is desirable to measure H_m by the means described in the preceding text or by a d-c. permeammeter method. From *B-H* curves obtained in this way it is possible to calculate apparent watts per pound with satisfactory accuracy for a sine-wave variation of induction. It must be emphasized here that the a-c. method of measuring H_m herein described, where $l_m = 88$ cm., is particularly designed for use above the knee of the *B-H* curve and loses accuracy at lower inductions, where the reluctance of the corners of the test square cannot be neglected.[3]

There is a further source of error in measuring a-c. permeability that should not be overlooked. In deriving Eq. 12 (see Appendix I),[8] the self inductance of the secondary of the mutual inductance is assumed to be negligible relative to the resistance, R_H. It is not easy to determine the error introduced by neglecting this self inductance because it also depends upon the wave shape of the exciting current. The dependence is such that a sharply peaked current wave, such as is encountered in these tests, may greatly increase the error over what would be expected with a sinusoidal current. The most sharply peaked current wave and therefore the greatest error which could be encountered in the a-c. permeability test occurs when the flux in the sample varies sinusoidally. It is shown in Appendix I that for this condition the error in the measured value of the peak current will be approximately

$$\text{Error in peak current} = \tfrac{1}{2}\,\frac{B_m}{H_m}\left(\frac{dH}{dB}\right)_{H_m} \cdot \left(\frac{2\pi f L_2}{R_H}\right)^2 \ldots\ldots\ldots\ldots (17)$$

where

$\left(\frac{dH}{dB}\right)_{H_m}$ = the rate of change of H with respect to B at the maximum value of H,

f = fundamental frequency, and

L_2 = the self-inductance of the secondary of the mutual inductor.

For commercial materials now available $\frac{B_m}{H_m}\left(\frac{dH}{dB}\right)_{H_m}$ will rarely be greater than 20, so that Eq. 17 becomes:

$$\text{Error} = 10\left(\frac{2\ \pi f L_2}{R_H}\right)^2$$

Thus for the mutual inductor described earlier for use with the 700-turns test frame where $L_2 = 84.5$ millihenries and $R_H = 2000$ ohms, the maximum error in measuring peak exciting current due to secondary self inductance will be 0.25 per cent.

Clamping:

In the standard Epstein test it is necessary to clamp the test specimen at the corners to maintain a good butt joint. In tests where the double-lapped joint is used, it has been found that strains due to the clamping more than offset any advantage gained by the clamping. In the opinion of the author, elimination of the corner clamps is one of the advantages of the double-lapped joint test, since it reduced by one the uncontrolled factors in the test.

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[8] See p. 794.

Design Considerations:

In setting up a test such as has been described, many of the constants can be selected in such a way as to simplify necessary calculations. For this reason a 700-turn test frame seems to be most convenient. The author's use of 352 turns was dictated in part by the test instruments available and is considerably less convenient than 700 turns, particularly for silicon iron with a specific gravity of 7.5.

The value of mutual inductance of inductor M, Fig. 1, was chosen to make R_H equal to 2000 or 20,000 ohms for a desirable calibration of the d-c. milliammeter. These are minor considerations, although they do have some value in avoiding errors in routine testing.

CONCLUSION

In the preceding text an attempt has been made to describe an apparatus and method of testing for a-c. core loss and a-c. permeability, together with some of the method's limitations, in sufficient detail to permit the setting up and intelligent use of such an apparatus. Very little has been said of the advantages of this test as compared to the standard tests. Most of the advantages are obvious, and so have not been mentioned. One which perhaps should be emphasized is the reproducibility of results, although the absolute accuracy of the test results is probably no better than with the standard methods.

APPENDIX I

The relation between R_H, Fig. 1, and the calibration of the d-c. milliameter in terms of H_m, as expressed in Eq. 12 may be derived as follows:

Instantaneous voltages are summed around the secondary circuit of the mutual inductor, M, Fig. 1.

$$M\frac{dI}{dt} + L_2\frac{dI_2}{dt} + R_H I_2 = 0 \quad . . (12a)$$

where M = mutual inductance of M,
L_2 = self inductance of the secondary of M,
I = primary current of M, and
I_2 = secondary current of M.

The reading, $I_{d.c.}$, of the d-c. milliameter may be expressed by

$$I_{d.\,c.} = 2f\int_0^{\frac{1}{2f}} I_2\,dt, \quad . . . . . (12b)$$

where initial time $t = 0$ occurs at $I_2 = 0$. Then substituting the value of I_2 from Eq. 12a in Eq. 12b

$$I_{d.\,c.} = -\frac{2f}{R_H}\int_{t=0}^{t=\frac{1}{2f}} M\,dI - \frac{2f}{R_H}\int_{t=0}^{t=\frac{1}{2f}} L_2\,dI_2 \quad . . (12c)$$

At the integration limits $t = 0$ and $t = \frac{1}{2f}$, $I_2 = 0$ by definition. Therefore

$$\int_{t=0}^{t=\frac{1}{2f}} L_2\,dI_2 = 0 \quad . . . . . (12d)$$

The integration limits of the other right-hand member of Eq. 12c are more difficult to evaluate, so for the moment let us assume that $I = I'$ when $t = 0$ and $I = -I'$ when $t = \frac{1}{2f}$, then Eq. 12c becomes

$$I_{d.c.} = \frac{4fMI'}{R_H} \quad . . . . (12e)$$

It is apparent that the meter reading, $I_{\text{d.c.}}$, is actually proportional to I', the value of primary current at the instant when $I_2 = 0$, rather than I_m, the peak value which we wish to measure. However, if the ratio of L_2 to R_H is made to approach zero, the time interval between I_m and I' will also approach zero, so that the value of I' will approach that of I_m and $\left(\dfrac{I_m - I'}{I_m}\right)$ can be made less than the required precision of measuring I_m. Therefore, we may substitute I_m for I' in Eq. 12*e*, provided that the ratio of L_2 to R_H permits satisfactory precision, and Eq. 12*c* becomes

$$I_{\text{d.c.}} = \frac{4fMI_m}{R_H} \ldots\ldots\ldots (12f)$$

In terms of magnetizing force

$$I_m = \frac{H_m l_m}{0.4\pi N_1} \ldots\ldots\ldots (12g)$$

Substituting for I_m and solving for R_H in Eq. 12*f*

$$R_H = \frac{10fMl_m}{\pi N_1}\cdot\frac{H_m}{I_{\text{d.c.}}} \ldots\ldots\ldots (12)$$

In addition to deriving Eq. 12 it is desirable to attempt to derive an expression for the error in measuring H_m in terms of L_2 and R_H. The percentage error in H_m, (ϵ), is equal to the percentage error in I_m, or

$$\epsilon = \frac{I_m - I'}{I_m} \ldots\ldots\ldots (17a)$$

The exciting current wave tends to be peaked for these tests, so that for an equal time interval between I_m and I', the error will be larger than for a sinusoidal current wave and the more sharply peaked the wave the greater the error.

When magnetizing an iron core from a sinusoidal voltage source. the wave shape of the flux in the core and consequently the wave shape of the exciting current depends upon the impedance in series with the core. Thus with zero series impedance, the flux is sinusoidal and the current sharply peaked. When series inductance or resistance is introduced, both the flux and current waves broaden until in the limit the current becomes sinusoidal. Since there is no satisfactory method at present for calculating the wave form of the exciting current for other than the limiting cases, we must select for purposes of this calculation the limiting case where the error in H_m is largest or where the flux is sinusoidal and the current wave is peaked. This will be on the safe side, however, as it makes the calculated error greater than will ever actually be encountered.

However, if for the moment we consider the current to be sinusoidal, the time interval between $I = I_m$ and $I_2 = 0$ or $I = 0'$ is

$$\Delta t = \frac{1}{2\pi f}\tan^{-1}\frac{2\pi fL_2}{R_H} \ldots\ldots (17b)$$

Now since the angle will be very small, we may assume that the tangent of the angle equals the angle or

$$\Delta t = \frac{L_2}{R_H} \ldots\ldots\ldots (17c)$$

so that under these conditions it can be seen that the time interval will be the same regardless of the frequency. Therefore, if the current I may now be expressed in a Fourier's series, the time interval will be the same for each harmonic, and $\dfrac{L_2}{R_H}$ may be regarded as the time interval between I_m and I'.

Since the flux density B in the test sample varies sinusoidally, the change, ΔB, in the time interval, Δt, is

$$\begin{aligned}\Delta B &= B_m\left[1 - \sin\left(\pi/2 + \frac{2\pi fL_2}{R_H}\right)\right]\\ &= \frac{1}{2}B_m\left(\frac{2\pi fL_2}{R_H}\right)^2, \text{ approximately} \ldots (17d)\end{aligned}$$

or the change ΔH in H is approximately

$$\begin{aligned}\Delta H &= \Delta B\left(\frac{dH}{dB}\right)_{H_m}\\ &= \frac{1}{2}B_m\left(\frac{dH}{dB}\right)_{H_m}\left(\frac{2\pi fL_2}{R_H}\right)^2 \ldots (17e)\end{aligned}$$

Therefore

$$\epsilon = \frac{\Delta H}{H_m} = \frac{1}{2}\frac{B_m}{H_m}\left(\frac{dH}{dB}\right)_{H_m}\left(\frac{2\pi fL_2}{R_H}\right)^2 \ldots (17)$$

DISCUSSION

Mr. Thomas Spooner.[1]—Some years ago Committee A-6 on Magnetic Properties developed a low induction test using the 25-cm. sample. This was intended primarily to be used in connection with core material for telephone and radio apparatus. It worked out very successfully and is used by a number of laboratories. After the development of this test, the 25-cm. sample proved to be so convenient that it was a logical step to develop its use for high inductions, and this work that Mr. Burgwin has described today is the outcome of that extension of the use of the 25-cm. sample. We are making a series of round-robin tests in the committee at the present time, and hope—it is only a hope because of delays due to war activities—that those tests may be carried far enough so that we can present a tentative method of test to the Society next year. Mr. Burgwin has done a very complete job in describing his test methods, experiences, and difficulties, and it is going to be a very simple matter to convert his paper into a test specification. I am sure that the committee is very grateful to him for the very detailed report he has given of his work.

Mr. S. L. Burgwin[2] (*author's closure*).—There is not much that I can add to Mr. Spooner's remarks, which seem to me to sum up the situation in regard to the 25-cm. Epstein test very nicely, except to thank Mr. Spooner and Committee A-6 for the support and encouragement which they have given this development.

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FATIGUE AND STATIC LOAD TESTS OF A HIGH-STRENGTH CAST IRON AT ELEVATED TEMPERATURES

By W. Leighton Collins[1] and James O. Smith[1]

Synopsis

Repeated-load and short-time static-load tests of unnotched and notched specimens cut from large plates of one heat of a commercial high-strength cast iron are reported. Very little difference between the static ultimate strengths of unnotched and notched (circumferential V-groove) specimens could be observed at any temperature from room temperature to 1100 F. The endurance limits for unnotched and notched, small transverse hole, specimens for completely reversed cycles of bending stress were determined for seven temperatures ranging from room temperature to 1200 F. The endurance limits for both types of specimens were practically equal for the range of temperatures used, provided the net cross-section is used in computing the stresses in the specimens with the holes. The ratio of endurance limit to static tensile strength had an almost constant value of 0.44 for the entire range of temperature.

The repeated-load and static-load tests at elevated temperatures reported in this paper are part of an extensive investigation of the mechanical properties of a high-strength cast iron. Short-time static-load tension tests were made at temperatures ranging from room temperature to 1100 F. The results of these tests helped in planning the fatigue tests at elevated temperatures and are considered to be of practical value in determining attainable strengths at various temperatures under extreme overload conditions occurring for a short period of time. However, for many uses at elevated temperatures long-time static tension (time-to-fracture) tests are highly desirable and are, therefore, included in the future plans of the investigation. Since cast iron is used in some cases at high temperatures in machine parts having stress concentrations as well as in parts free from stress concentrations, both notched and notch-free specimens were used in the static and fatigue tests. The repeated-load tests may in general be more directly useful than the tensile strengths in that modern service conditions in steam turbines, pumps, etc., frequently subject cast iron to repeated loads at relatively high temperatures. Long-time static tests from which the creep properties may be obtained are also desirable but are not discussed in this paper.

Material and Test Specimens

The material used came from one pouring of a high-strength cast iron furnished by the Gardner-Denver Co.

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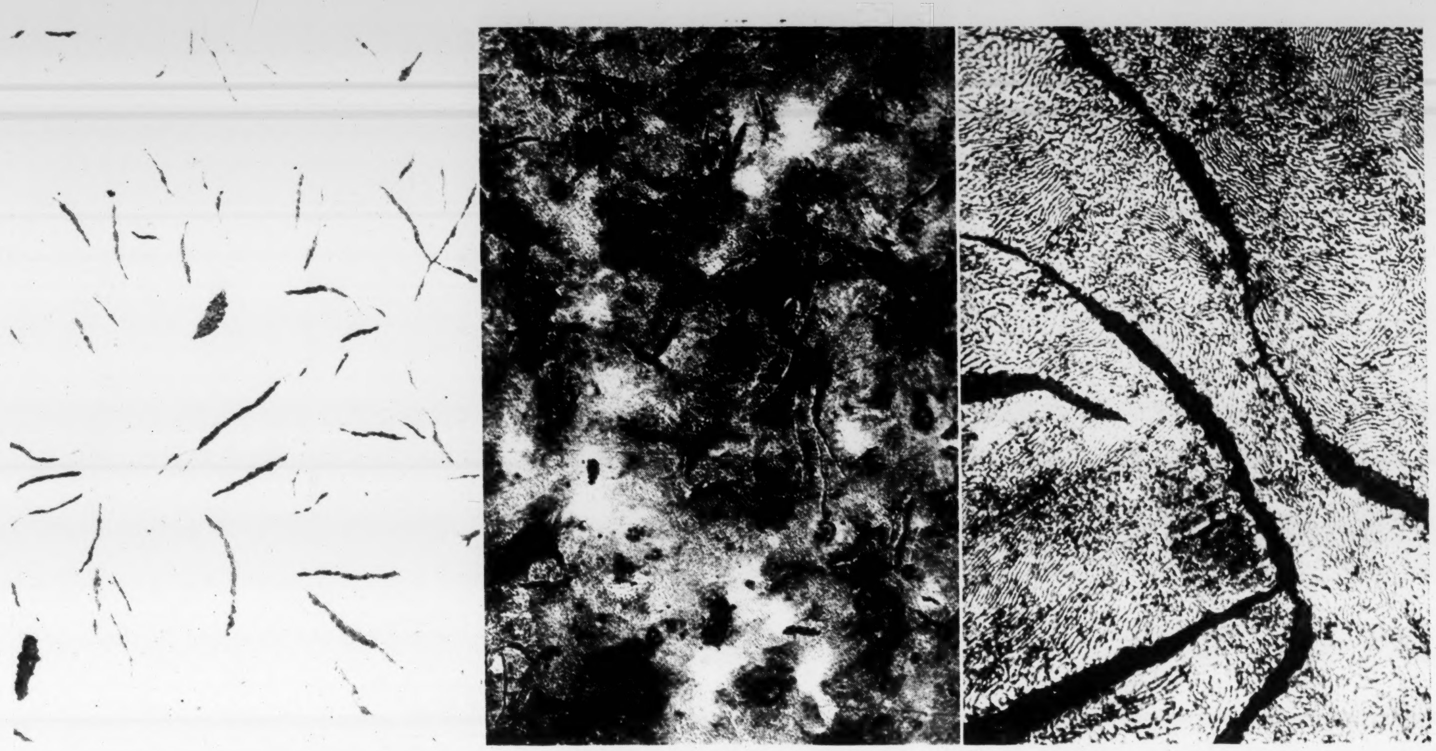


(*a*) Unetched (X 100). (*b*) Etched in 2 per cent Nital (X 100). (*c*) Etched in 2 per cent Nital (X 500).

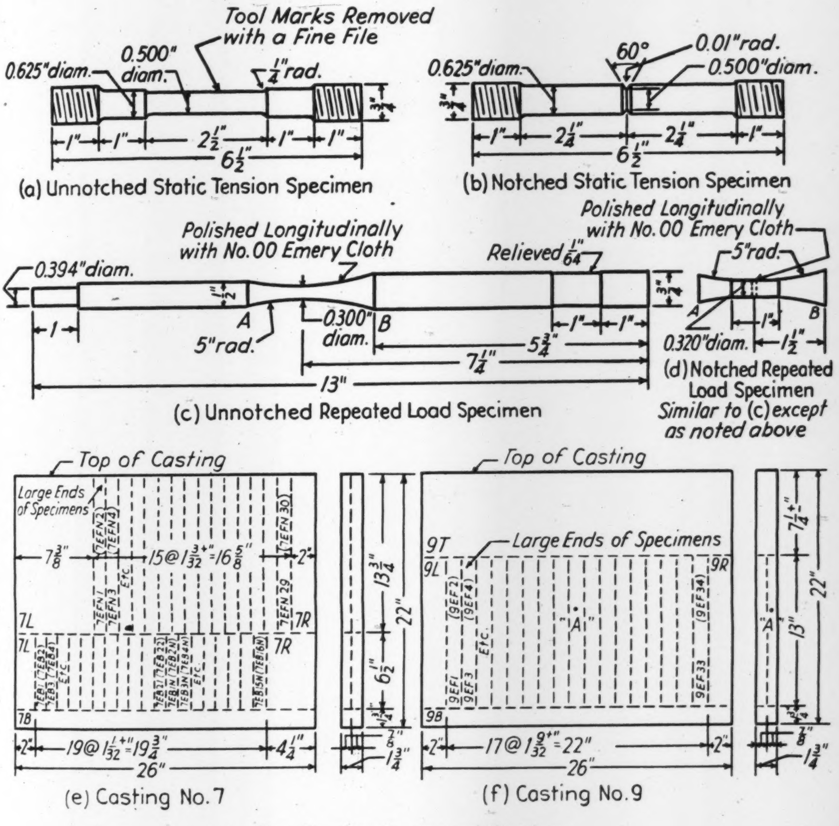
FIG. 1.—Typical Photomicrographs.

Specimens taken from one-quarter point of the thickness near center of casting.

of Quincy, Ill. The iron was a "process iron" made by the Sarbo-Mat Process. The furnace charge consisted of 78 per cent low carbon steel scrap, 21 per cent factory and foundry scrap and 1 per cent manganese; the Sarbo-Mat alloy was added at the cupola spout. The castings were in the form of large slabs, cast vertically and poured from the top, 26 in. long, $1\frac{3}{4}$ in. thick, and 27 in. high. All material used was in the "as cast" condition.

The chemical composition as determined from a specimen taken from the center of casting No. 3, not used for these tests but the location is shown as point *A* in Fig. 2(*f*), is as follows:

Total carbon, per cent	2.84
Combined carbon, per cent	0.74
Graphitic carbon, per cent	2.10
Silicon, per cent	1.52
Manganese, per cent	1.05
Copper, per cent	0.37
Chromium, per cent	0.31
Nickel, per cent	0.20
Molybdenum, per cent	0.00
Phosphorus, per cent	0.07
Sulfur (evolved as H_2S), per cent	0.106
Sulfur (gravimetric), per cent	0.124


Tool Marks Removed with a Fine File
0.500" diam.
0.625" diam.
1/4" rad.
3/4"
1" 1" 2 1/2" 1" 1"
6 1/2"
60°
0.01" rad.
0.500" diam.
0.625" diam.
3/4"
1" 2 1/4" 2 1/4" 1"
6 1/2"
Polished Longitudinally with No. 00 Emery Cloth
0.394" diam.
1/2"
A
5" rad.
0.300" diam.
B
Relieved 1/64"
1" 1"
3/4"
1
5 3/4"
7 1/4"
13"
Polished Longitudinally with No. 00 Emery Cloth
5" rad.
A B
1"
0.320" diam.
1 1/2"
Top of Casting
Large Ends of Specimens
7 3/8"
15 @ 1 3/32" = 16 5/8"
2"
7L
7R
7B
7EFN2
7EFN4
7EFN1
7EFN3
ETC.
7EFN 30
7EFN 29
7EB1
7EB3
7EB2
7EB4
7EB32
7EB31
7EB1N
7EB3N
7EB2N
7EB4N
7EB5N
7EB6N
2" 19 @ 1 1/32" = 19 3/4" 4 1/4"
26"
13 3/4"
22"
6 1/2"
1 1/4"
7/8"
1 3/4"
Top of Casting
Large Ends of Specimens
9T
9L
9R
9B
9EF2
9EF4
9EF1
9EF3
ETC.
9EF34
9EF33
A
2" 17 @ 1 9/32" = 22" 2"
26"
7 1/4"
13"
22"
1 3/4"
7/8"
1 3/4"

(a) Unnotched Static Tension Specimen

(b) Notched Static Tension Specimen

(c) Unnotched Repeated Load Specimen

(d) Notched Repeated Load Specimen Similar to (c) except as noted above

(e) Casting No. 7

(f) Casting No. 9

FIG. 2.—Specimens and Castings.

Typical photomicrographs from location *A* of casting No. 3 are shown in Fig. 1. The material photographed was half-way between the outside and center of the thickness of the casting, thus being representative of the material at the center of the test specimens used in this investigation.

The four types of specimens used, their dimensions, and their locations in the castings are shown in Fig. 2. Specimen *a* is in accordance with the A.S.T.M. Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 37 T)[2] and was used for the unnotched short-time static tension tests at all temperatures. Specimen *b* is the notched (circumferential V-groove) specimen tested similarly to *a* in order to determine notch sensitivity under short-time static tensile loads at various temperatures. Specimens *c* (unnotched) and *d* (notched with a transverse, diametral hole) were used for the repeated-load tests at elevated temperatures. The endurance limits at room temperature were determined from specimens having critical sections similar to specimens *c* and *d* but which were tested in rotating-beam, Farmer type machines. It will be noted, from Fig. 2(*e*) and (*f*), that the location of each specimen in the casting is known. The first number for each specimen designates the number of the casting from which the specimen came; the other numbers and letters indicate type of test, location in casting, etc.

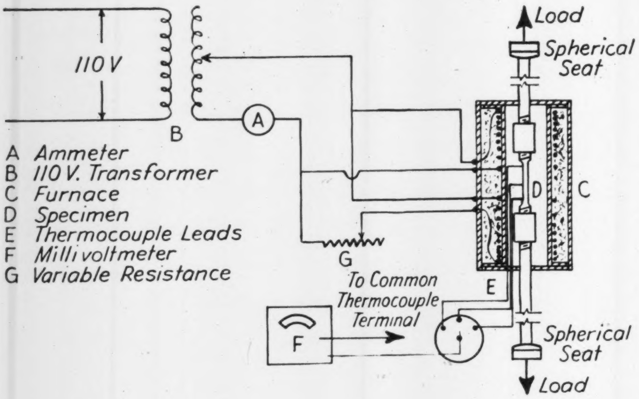


FIG. 3.—Diagrammatic Sketch of Furnace and Wiring for Short-Time Tension Tests.

EQUIPMENT AND TEST PROCEDURE

Static Tests:

The static-load tests were made in a 10,000-lb. motor-driven Olsen screw-power testing machine whose accuracy is within the permissible variation allowed by the A.S.T.M. Standard Methods of Verification of Testing Machines (E 4 – 36).[3] The free speed of loading head used was 0.075 in. per min. for unnotched specimens and 0.018 in. per min. for the specimens with notches; thus the time of testing (period during which load was applied) for both types of specimens normally varied from 6 to 8 min., the shorter times usually being for the higher temperatures. Only the maximum load supported by each specimen was recorded.

A diagrammatic sketch of the apparatus used for the short-time tension tests of unnotched specimens at elevated temperatures is shown in Fig. 3. For the notched - specimens the furnace coils were connected in series in order to reduce the amount of current and in order to use a Leeds and Northrup potentiometer recorder and controller; a shunt was inserted in the top coil of the furnace in order to maintain the permissible temperature gradient, but its insertion was found unnecessary; and a double-pole switch was inserted at *E* of Fig. 3 in order that a Micromax recorder could be used as a check on the temperature. However, only one recorder was in the circuit at the time of reading the temperature, which was always determined from a millivoltmeter and a Leeds and Northrup conversion table. The calibration of the thermocouples used showed such practice to be feasible.

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[2] 1939 Book of A.S.T.M. Standards, Part I, p. 1254.

[3] *Ibid.*, p. 767.

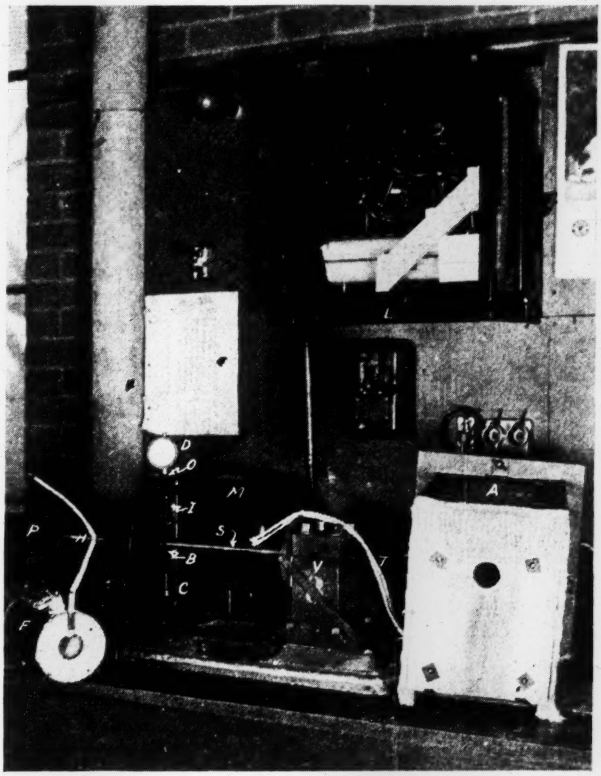

FIG. 4.—Rotating-Spring Fatigue Testing Machine.

The furnace was so constructed that a temperature gradient of ten degrees or less over the length between threaded ends, conforming with E 21 - 37 T, was obtained. Maintenance of this gradient was checked for each specimen by the use of at least three thermocouples, one at the center of the turned-down length and one near each fillet. The reading of the center thermocouple, at the root of the notch for the notched specimens, was always used as the temperature of the specimen.

In all cases the specimens were slowly brought to temperature in 2 to 2½ hr. by a current not exceeding by more than 25 per cent that required to maintain the test temperature. The test temperature was normally maintained within ±5 to ±7 F. for at least 30 min. prior to starting the application of load, thus making certain of thermal equilibrium.

Fatigue Tests:

The repeated-load tests at elevated temperatures were made in two rotating-spring fatigue testing machines[4] designed in the Fatigue Laboratory of the University of Illinois and operating at speeds of 2500 rpm. This type of constant strain machine was used because the specimen is stationary and its temperature can thus be measured accurately by means of attached thermocouples. The machine is shown in Fig. 4. The large end of specimen *S* is held rigid in the vise *V*, and the other end which runs in roller bearing *B* is rotated in a small circle by means of shaft *H* and pulley *P* which is driven by an electric motor *M*. Consequently the specimen is subjected to completely reversed cycles of bending stress. The stress in the specimen is governed by the compression of the calibrated spring *I*, the compression being measured, when the machine is stopped, by means of a removable dial *D* with a long plunger inserted in hole *O*. After proper adjustment of load, the deflection was kept constant by means of clamp *C*. The electric furnace *F* is placed around the specimen, *S*, and is attached to a Leeds and Northrup potentiometer recorder and controller *L*. During a test the furnace is covered with a thick-walled asbestos box *A*. The thermocouples used to measure the temperature of the specimen are marked *T*.

At frequent intervals during a test the machine was stopped, the clamp loosened and the load checked. In this manner the load was kept practically constant throughout the entire test. In all cases the specimen was brought nearly to the test temperature by a current slightly in excess of that required to maintain the temperature desired. The current was then reduced and the specimen reached the test temperature by the time loading was completed. At the higher temperatures great care was taken to prevent creep from causing any of the measured deflection. One division of the dial on one machine represented a maximum stress of 166 psi. at the smallest section of the specimen; the other machine had a similar constant. The normal variation during a test was 0.5 divisions and never exceeded 3 divisions.

The furnace was calibrated and adjusted so that the temperature variation from the mean over a 1-in. length containing the critical section of the specimen did not exceed ±5 F. in most cases and in no case exceeded ±10 F. The temperature of the furnace was maintained within a range of ±7 F.; the No. 12 wire chromel-alumel thermocouple leading to the recorder and controller was placed near the inside wall of the furnace to provide the most sensitive control. The temperature of the specimen was determined by means of a calibrated chromel-alumel thermocouple made of No. 34 wires carefully attached to the specimen at its smallest section. In each test a second thermocouple was placed at a distance of 0.5 in. from the smallest section of the specimen in order to check the temperature gradient. The temperature variation of the specimen at each thermocouple rarely exceeded a range of ±3 F. during the progress of a test.

TEST DATA AND RESULTS

Static Tests:

The tensile strengths determined from the short-time tests are shown in Fig. 6, the stress for the notched specimens being based on the area at the root of the groove. Although the tensile strength values for the notched specimens are frequently slightly less than the values for unnotched specimens, the differences in almost all cases are less than the variation between several values for the same type of specimen tested at the same temperature. Consequently it is felt that no significant difference is observed between the strengths of unnotched and notched (circumferential V-groove) specimens for temperatures in the range from room temperature to 1100 F.

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[4] Fully described by H. F. Moore and G. N. Krouse, "Repeated Stress (Fatigue) Testing Machines Used in the Materials Testing Laboratory of the University of Illinois," *Circular No. 23*, Engineering Experiment Station, University of Illinois (1934); also H. F. Moore and N. J. Alleman, "Progress Report on Fatigue Tests of Low-Carbon Steel at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 114 (1931).

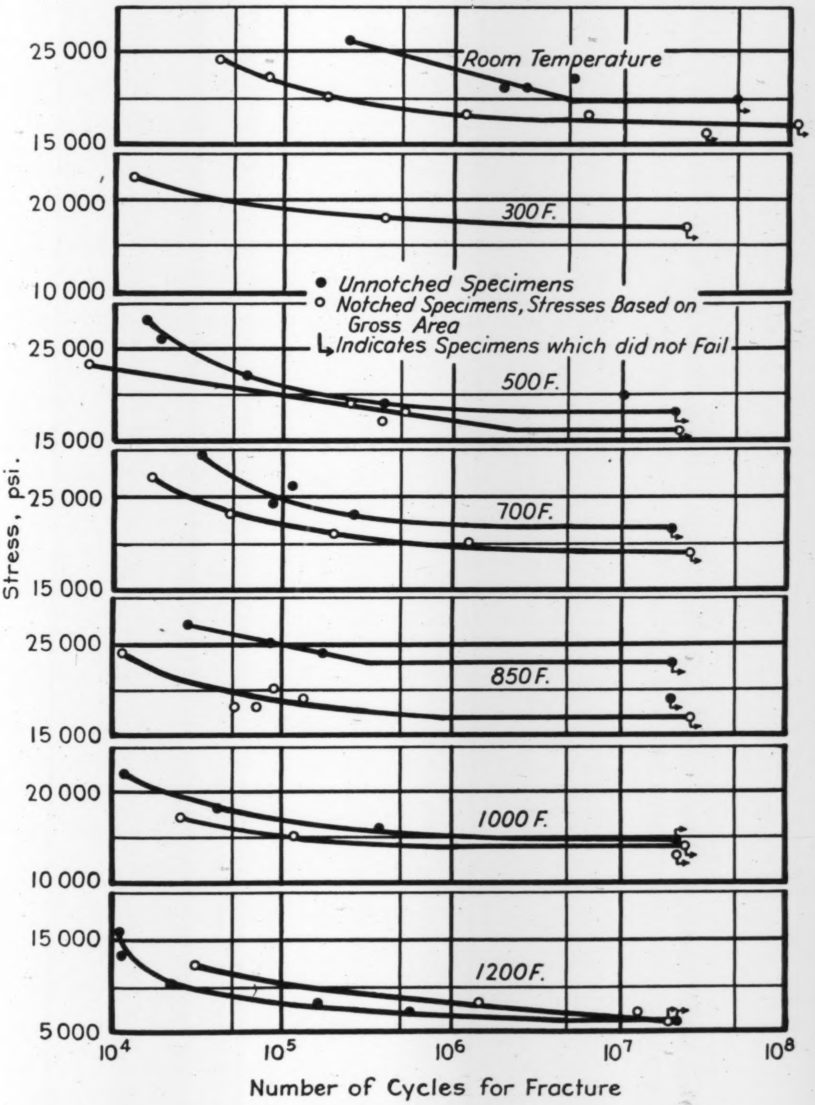

FIG. 5.—*S-N* Diagrams for Completely Reversed Cycles of Bending Stress.

Fatigue Tests:

The *S–N* (stress-number of cycles to failure) curves for the repeated-load tests at all temperatures used are shown in Fig. 5 and the endurance limits[5] are tabulated in Table I. It should be noted that the stresses used for the notched specimens are based on the gross area, no allowance having been made for the material removed in forming the hole. All endurance limits are based on at least 20,000,000 cycles of stress and in all but one case the difference between the stress in the last specimen that broke and the endurance limit did not exceed 1000 psi. It will be observed that the endurance limits for the notched (0.032-in. diameter hole) specimens are only slightly less than for the unnotched specimens at all temperatures except 1200 F., and there the endurance limits have a common value. Attention is called to the fact that specimens tested at 3000 to 4000 psi. above the endurance limits broke at relatively small numbers of cycles; this means that any overloading, even though it is for a relatively small number of cycles, may lead to an early failure.

TABLE I.—ENDURANCE LIMITS.

Temperature, deg. Fahr.	Endurance Limits, psi.		
	Unnotched	Notched[a]	
		Gross Area	Net Area
Room	20 000	17 000	20 400
300	....	17 000	20 400
500	18 000	16 000	19 200
700	21 600	19 000	22 800
850	23 000	17 000	20 400
1000	14 500	14 000	16 800
1200	6 000	6 000	7 210

[a] Notch consists of a transverse hole whose diameter is 0.10 the diameter of the specimen.

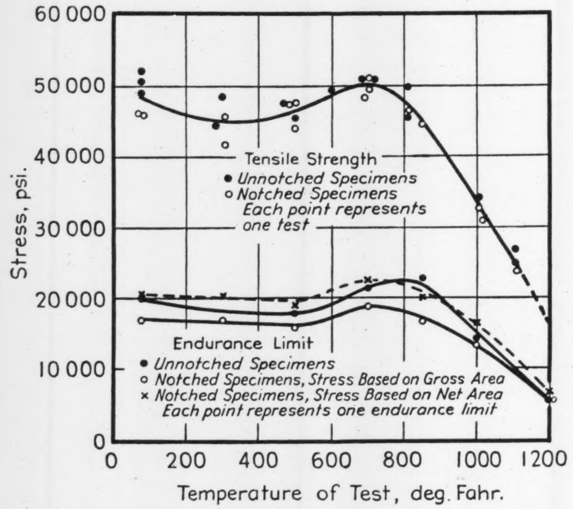

FIG. 6.—Strength-Temperature Curves.

The endurance limits plotted against temperature are shown in the lower part of Fig. 6. It should be noted that here the endurance limits for the notched specimens are plotted using both the gross and the net cross-sections in computing the stresses. A comparison of the data for the unnotched and the notched specimens can be made even though the specimens did not come from the same casting. From data not included in this report a variation of 3900 psi. was found in the tensile strengths of similarly located specimens in three castings. This variation is no greater than the variations found for strengths of specimens taken from casting No. 7 and tested at room temperature (see Fig. 6). Thus a comparison of the endurance limits for the unnotched and notched specimens can be made without regard for any significant difference in the material of the two castings from which the specimens came.

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[5] Although the terms "endurance limit of unnotched specimens" and "endurance limit of notched specimens" are used in this report, it should be noted that the endurance limit of the material is obtained from the tests of unnotched specimens.

The shapes of the temperature - endurance limit curves are quite similar to the shape of the temperature - tensile strength curve except for what appears to be an unusually high value of endurance limit for unnotched specimens at 850 F. This causes the "hump" in the curve for endurance limits to be at about 800 F. whereas the greatest tensile strength was obtained at 700 F. The fact that the curve for endurance limits based on net areas coincides so closely with the curve for the unnotched specimens indicates that the stress concen-

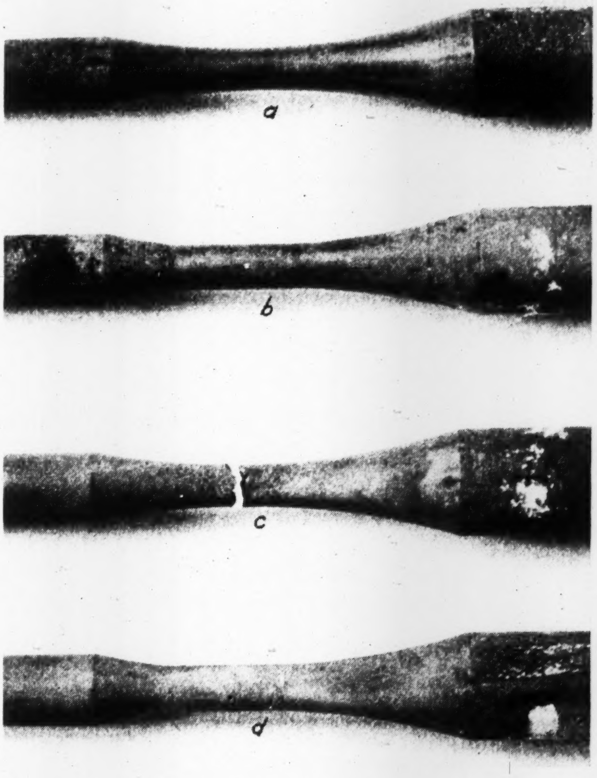

FIG. 7.—Fatigue Specimens Showing Scaling at Elevated Temperatures.

(*a*) Specimen No. 9EF18—20,028,100 cycles at 850 F.
(*b*) Specimen No. 7EFN11—25,547,400 cycles at 850 F.
(*c*) Specimen No. 9EF32—21,164,800 cycles at 1200 F.
(*d*) Specimen No. 7EFN21—20,000,000 cycles at 1200 F.

tration factor is unity for all temperatures used.

The endurance ratios, which are the average values of the endurance limits of unnotched and notched, based on net cross-section, specimens for any temperature divided by the average tensile strength of all specimens tested at that temperature, are listed in Table II. An average value of 0.44 is obtained. The value of tensile strength at 850 F. given in Table II is interpolated from the curve in Fig. 6; the value for 1200 F. is extrapolated from the values up to 1100 F. The maximum variation from the average value of the endurance ratio is ±9 per cent. The practically constant value of this ratio leads to the belief that the material was relatively metallurgically stable at all temperatures used and for the time involved in the tests.

TABLE II.—COMPARISON OF ENDURANCE LIMITS AND TENSILE STRENGTHS.

$$\text{Endurance ratio} = \frac{\text{Average endurance limit at a given temperature}}{\text{Average tensile strength at the same temperature}}$$

Temperature, deg. Fahr.	Average Endurance Limit, psi.[a]	Average Tensile Strength, psi.	Endurance Ratio
Room	20 200	48 400	0.42
300	19 400	45 000	0.43
500	18 600	46 500	0.40
700	22 200	50 200	0.44
850	21 700	44 800	0.48
1000	15 700	33 200	0.47
1200	6 600	16 500	0.40[b]
		Average =	0.44

[a] Average of endurance limits of unnotched specimens and of notched specimens, based on net cross-section.

[b] Extrapolated from 1100 F.

The specimens subjected to high temperatures for long periods of time, maximum of about 6 days, in the fatigue tests underwent slight scaling. This condition is shown in Fig. 7. The scaling may account for a small amount of the decrease in endurance limits at temperatures above 800 F., but in view of the fact that there was no scaling in the short-time tension tests and that the endurance ratio is so nearly constant for all temperatures (see Table II) the decrease apparently is accounted for by inherent weakening of the material due to the high temperature.

CONCLUSIONS

From a study of the data in the tables and of the figures, the following conclusions regarding the tests of a high-strength cast iron at elevated temperatures can be reached:

Short-Time Static Tension Tests:

1. A circumferential V-groove had very little effect on the tensile strength at temperatures ranging from room temperature to 1100 F. In other words, the stress concentration factor under these conditions was practically unity.

2. The average tensile strength of all specimens, notched and unnotched, varied from 49,000 psi. at room temperature to 45,000 psi. at 300 F., to a maximum of 49,500 psi. at 700 F., and to only 26,000 psi. at 1100 F.

Fatigue Tests in Bending:

3. A transverse hole had no effect upon the fatigue strength of the high-strength cast iron tested for any temperature ranging from room temperature to 1200 F., provided that the stresses in the specimens containing the holes are calculated on the basis of the net cross-section at the holes.

4. The endurance limit for completely reversed cycles of bending stress as determined from unnotched and notched specimens, based on net cross-section, varied from 20,200 psi. at room temperature to 19,500 psi. at 300 F., to a maximum of 22,500 psi. at 800 F., and to 6500 psi. at 1200 F.

5. The endurance ratio, that is, the ratio of the endurance limit for completely reversed cycles of bending stress to the tensile strength, has an almost

constant value of 0.44 for temperatures ranging from room temperature to 1200F.

6. The maximum static and fatigue strengths occurred at approximately 700 F. and both strengths decreased rapidly at higher temperatures.

Acknowledgments:

All of the work herein reported was carried out as a project of the Engineering Experiment Station in the Department of Theoretical and Applied Mechanics of the University of Illinois. Valuable assistance was given by O. C. Worley, Research Graduate Assistant, in making the repeated-load tests and the short-time tension tests of notched specimens, and by T. E. McVay, senior student in Mechanical Engineering, in making the short-time tests of unnotched specimens. The furnace was designed and built by H. R. Puckett, Research Graduate Assistant, under the direction of T. J. Dolan, Assistant Professor. Acknowledgment is made to J. T. MacKenzie of the American Cast Iron Pipe Co. for his continued interest and courtesy in providing the chemical analysis and the photomicrographs.

DISCUSSION

Mr. J. S. Vanick[1] *(presented in written form)*.—By establishing the fact that the ratio between fatigue strength and static strength of a cast iron adheres to a fixed ratio and follows a parallel course from room temperatures to 1100 F., the authors have established an important relationship which up to this time had been taken for granted but not proven. There is further satisfaction in the discovery that notched bars retain practically the full strength of the material for the net cross-section. In addition to the applications to which the authors have called attention, their results should be helpful in working out the design of castings such as piston rings and engine cylinder sleeves, pistons, and heads, which in many cases are subject to repeated loads at relatively high temperatures. The fatigue strength at low and high temperatures of the harder, low-alloyed heat-resisting irons would suggest an interesting study for the extension of this work.

The authors are aware that their tests are applicable only to iron that has been exposed to loading at elevated temperatures for a relatively short time. This point is re-emphasized here because a lower-strength cast iron made from a composition possessing better heat resistance than the material tested might be able to retain its initial strength and presumably its fatigue strength at elevated temperatures over a much longer period of time than a stronger iron of the 50,000 or 60,000 psi. tensile strength class which possesses less resistance to deterioration by heat. It is known, for example, that steam at temperatures of 700 to 900 F. is much more destructive than muffle furnace atmospheres at these same temperatures, and a valuable contribution to the extension of the application of cast iron would result if this work inspires someone to establish the static and fatigue strengths of cast irons at temperatures in the 700 to 1200 F. range by testing a number of suitable compositions over a long (6 to 12 months) period of time.

Mr. Jasper O. Draffin[2] *(presented in written form)*.—In view of the large use of cast iron for stress-bearing machine parts, this investigation is most timely. It is apparent that great care was used by the authors to maintain constant temperatures, uniform throughout the specimens for each series of tests—a condition essential for good results.

The chemical analysis does not indicate any large amount of alloying elements in the iron, and the question may be asked as to how the high strength was obtained. It would also be desirable to know something of the hardness of this iron. With regard to the assumption of uniformity in the different castings, the difference seems to be about 8 per cent. Since tests on other irons have shown a variation as great as 12 to 14 per cent within a single casting and an average of a number of tests for a number of castings from a single heat has given as high as 10 per cent variation, the assumption seems justified.

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[1] Metallurgist, The International Nickel Co., Inc., Bayonne, N. J.

[2] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

Though the writer has made a few exploratory tests, in static torsion, of notched and unnotched specimens of ordinary gray cast iron without finding a significant difference in strength which could be attributed to the notch, he is somewhat surprised that no appreciable difference was found for similar tests in tension. Does this mean that stress concentrations due to sudden changes in cross-section do not lower the strength of brittle materials subjected to an axial tensile stress under static load?

While the reliability of the data is not questioned, the endurance limit for the unnotched specimens at a temperature of 850 F. looks out of line with the other values. It would be well to check these tests, if possible, since it may be that two unusually strong specimens were, by chance, tested at this temperature and stress. This check seems advisable, particularly in view of the fact that at this temperature the specimens failed at a relatively small number of repetitions of load when stressed but slightly above the endurance limit, a fact pointed out by the authors for all their tests.

It would be interesting to know whether the endurance ratio for ordinary gray iron agrees with that for high-strength iron at various temperatures.

MR. C. K. DONOHO[3] (*presented in written form*).—The authors of the paper have compared tests of unnotched and notched specimens and have adjudged that there is no significant difference in the tests attributable to the notching. There are useful statistical tests for significance which may be applied to these data to determine the level of probability of significance or nonsignificance of the effect of notching on the physical tests.

For example, the data of Table I for endurance limit of unnotched and notched (net area) bars may be analyzed by the method outlined by Fisher.[4]

Temperature, deg. Fahr.	Endurance Limits, psi. × 10^{-3}		Difference, Notched minus Unnotched, psi. × 10^{-3}
	Unnotched	Notched (Net Area)	
Room	20.0	20.4	0.4
300	....	20.4	...
500	18.0	19.2	1.2
700	21.6	22.8	1.2
850	23.0	20.4	−2.6
1000	14.5	16.8	2.3
1200	6.0	7.2	1.2
			$\bar{X} = +0.6167$

$$\frac{S^2}{6} = 0.475, \; t = \frac{0.6167}{\sqrt{0.475}} = 0.895$$

For $n = 5$, p is between 0.4 and 0.5.

Since the probability of the difference, p, occurring by chance is between 0.4 and 0.5, obviously the difference cannot be considered significant.

The results of the static tests shown in the upper part of Fig. 6 unfortunately are not given in numerical form and hence cannot be treated with exactness. However, the data were divided into groups by temperature intervals, the plotted points scaled off as accurately as possible, and the results of each kind of test for each group averaged to give values which may be considered as representative single tests to be compared.

Group	Temperature Interval, deg. Fahr.	Tensile Strength, psi. × 10^{-3}		Difference, Notched minus Unnotched, psi. × 10^{-3}
		Unnotched	Notched	
1	50 to 100	50.7	46	−4.7
2	270 to 320	46.5	44	−2.5
3	460 to 520	47	46.3	−0.7
4	650 to 750	51	49.3	−1.7
5	800 to 820	47.5	46	−1.5
6	990 to 1020	34	32	−2.0
7	1080 to 1120	26	24	−2.0
				$\bar{X} = -2.16$

$$\frac{S^2}{7} = 0.2237, \; t = \frac{2.16}{\sqrt{0.2237}} = 4.57$$

For $n = 6$, p is less than 0.01.

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[3] Metallurgist, American Cast Iron Pipe Co., Birmingham, Ala.

[4] R. A. Fisher, "Statistical Methods for Research Workers," Sixth Edition, p. 126, *et seq.*, Oliver and Boyd, London (1936).

It will be seen here that for the seven groups all of the differences are of the same sign, which is in itself a remote chance probability (2 in 128), while use of the calculated *t* shows that such a difference should occur by chance less than one time in 100 trials. In this case it appears that the differences are significant.

It is believed that the use of some such statistical method to estimate significance adds to the value and meaning of such data.

MESSRS. W. LEIGHTON COLLINS AND JAMES O. SMITH[4] (*authors' closure by letter*).—The authors wish to thank Mr. Vanick for mentioning the industrial applications wherein the data presented may be of value. The suggestions for continued work along similar lines are also appreciated and will serve as a guide for future work.

Mr. Draffin inquired about the hardness of the iron and the source of the high strength. The high strength, considering the small amounts of alloying elements present, is undoubtedly due to the fact that the material is a "process" iron, that is, the alloying is done at the cupola spout. Although no published reference can be given, the authors have been led to believe that for a given chemical composition a "process" iron will have a strength of about 10,000 psi. greater than an iron made by the more usual cupola method. The hardness of the iron, at location *A* of Fig. 2 (*f*), is 200 Brinell (3000-kg. load) and 15 on the Rockwell "C" sale.

No definite statement regarding Mr. Draffin's question about notch sensitivity of cast irons subjected to static loads will be made at this time. However, it is hoped that an answer will be available next year as the authors now have such an investigation for both static and repeated loads under way.

The statistical study of the data made by Mr. Donoho is a distinct contribution to the paper because it does present another method of interpreting the data. The authors are very grateful to Mr. Donoho for having made the study and submitting it as a discussion.

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[4] Assistant Professor, and Instructor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

THE STRAIN HARDENING OF GRAY CAST IRON

By John Sanford Peck[1]

Synopsis

The data presented in this paper show the effect produced upon the Rockwell hardness of gray cast iron by compressive loads of increasing intensity. Specimens of gray cast iron, 1 in. square and ½ in. high were used. The Rockwell B hardness of each specimen was obtained prior to loading by averaging 64 readings, 16 on each face. The specimens were then subjected to compressive loads, increasing by increments of 10,000 psi. and their Rockwell hardness obtained again. The change in hardness was computed and the results plotted. In order to investigate the possible effect of the height of the specimen on the results, a series of check tests was run using specimens 1, 1½, 2, and 3 in. in height, respectively. No material effect was observed.

The data show that up to about 40,000 psi. the Rockwell B hardness increases slightly. From 40,000 to 60,000 psi. no significant change in hardness was observed. From 60,000 psi. to failure the Rockwell B hardness decreased rapidly.

In order to explain this apparent violation of all the theories of strain hardening, micrographs were made of the same spot in one specimen after increasing compressive loads had been applied. These micrographs, taken in the polished and unetched condition, show minute cracks appearing after a load of 60,000 psi. and increasing after succeeding loads until failure.

It is concluded that the apparent softening of the metal as indicated by the differences in Rockwell hardness values taken before and after compressing is due to the penetrator of the Rockwell tester slipping into one of these minute fissures and giving a lower reading.

This somewhat informal study was undertaken in an effort to explain a phenomenon that appeared some two years ago in the course of student activities in the Materials Testing Laboratory of the College of the City of New York. At that time, the practice was instituted of requiring the students to make Rockwell hardness tests on all metallic specimens before and after test. The intention was to illustrate the phenomenon of strain hardening.

The results obtained were entirely consistent in all cases, with one notable exception. When cast iron was tested in compression, it was discovered that the Rockwell hardness number showed a marked decrease after test. Such a result is diametrically opposed to the amorphous metal hypothesis, the slip interference theory, or any other theory regarding strain hardening and it was felt that a study of the phenomenon might result in some explanation that would at least satisfy student curiosity and might possibly be of wider interest.

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[1] Assistant Professor of Civil Engineering, College of the City of New York.

It was realized at the start that when dealing with such a variable material as cast iron it would be necessary to take a large number of individual readings in order to obtain a mean value for the hardness of any one specimen that would approach reliability.

The specimens selected for the investigation were medium gray cast iron, 1 by 1 in. in cross-section and ½ in. high. Sixteen Rockwell hardness readings were taken on each 1 by ½-in. face, each reading in the center of an area ¼ by ⅛ in. Sixty-four readings thus constituted one set. The specimens were then loaded on their 1 by 1-in. faces and another set of hardness readings taken. The difference between the means of these two sets of hardness readings then gave the change in hardness due to the applied load. Separate specimens were used for each loading, the loads increasing by increments of 10,000 psi.

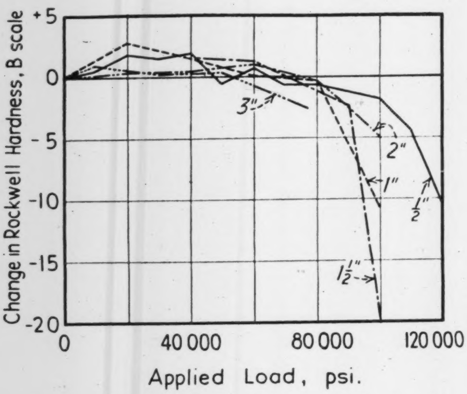


Fig. 1.—Effect of Compression on Rockwell Hardness of Gray Cast Iron.

For specimens of varying heights.

Specifically, the problem was to determine the relation between the applied compressive load and the change in hardness for gray cast iron. To this end twelve specimens 1 by 1 by ½ in. were tested as described above. The applied load was increased by increments of 10,000 psi.

In order to form some idea of the effect of the height of the specimen on the change in hardness, four additional series were run, using specimens 1, 1½, 2, and 3-in. high, respectively. In these check tests, the applied load was increased by increments of 20,000 psi.

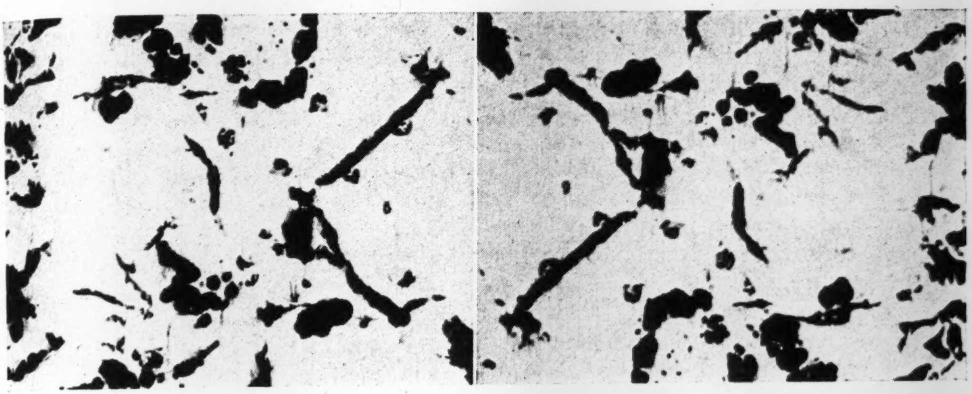
In Fig. 1 are plotted the data from the five series of tests, and a summary of the data is shown in Table I. An inspection of Fig. 1 shows that in the case of the ½-in. specimens increasing the applied load increases the hardness slightly up to a load of about 40,000 psi. From 40,000 to 60,000 psi., the load applied does not produce any significant increase in hardness over the original value, but beyond 60,000 psi. the applied load actually produces a decrease in hardness, and the rate of softening increases progressively as the point of fracture is approached.

TABLE I.—EFFECT OF COMPRESSION ON ROCKWELL HARDNESS OF GRAY CAST IRON.

Applied Compressive Load, psi.	Change in Rockwell Hardness, B Scale				
	½ in. High	1 in. High	1½ in. High	2 in. High	3 in. High
10 000	0.33	....	....	....	1.07
20 000	1.97	2.84	2.75	0.59	....
30 000	1.29	....	....	....	0.36
40 000	2.09	1.57	1.61	0.53	....
50 000	0.47	....	....	....	0.48
60 000	0.60	0.10	1.22	1.04	....
70 000	−0.62	....	....	....	−2.19
77 000	....	....	....	....	−2.46
80 000	−0.61	−0.26	−1.15	−0.23	....
90 000	−1.17	....	−2.24	....	....
100 000	−1.92	−10.97	−20.10	−5.64	....
110 000	−4.40	....	....	....	....
120 000	−10.21	....	....	....	....

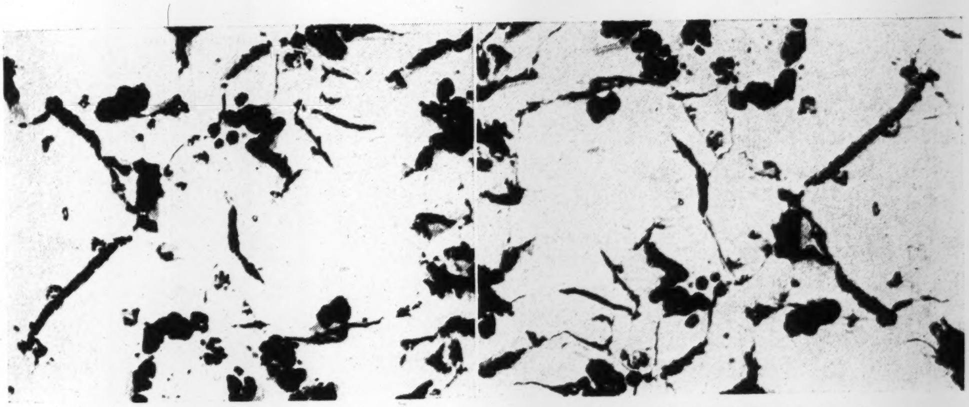
NOTE.—It is evident to anyone versed in statistical method that conclusions from such small differences between two means must be drawn with extreme caution, especially when dealing with a naturally variable material. The probabilities of all the above differences being due to pure chance (random errors of sampling) rather than to any constant factor have been computed and their significance determined. However, due to the exigencies of printing it was decided not to publish the results of the application of statistical method to these data. The author has all the supporting evidence and would be glad to submit it to anyone interested.

Figure 1 also shows that the only effect



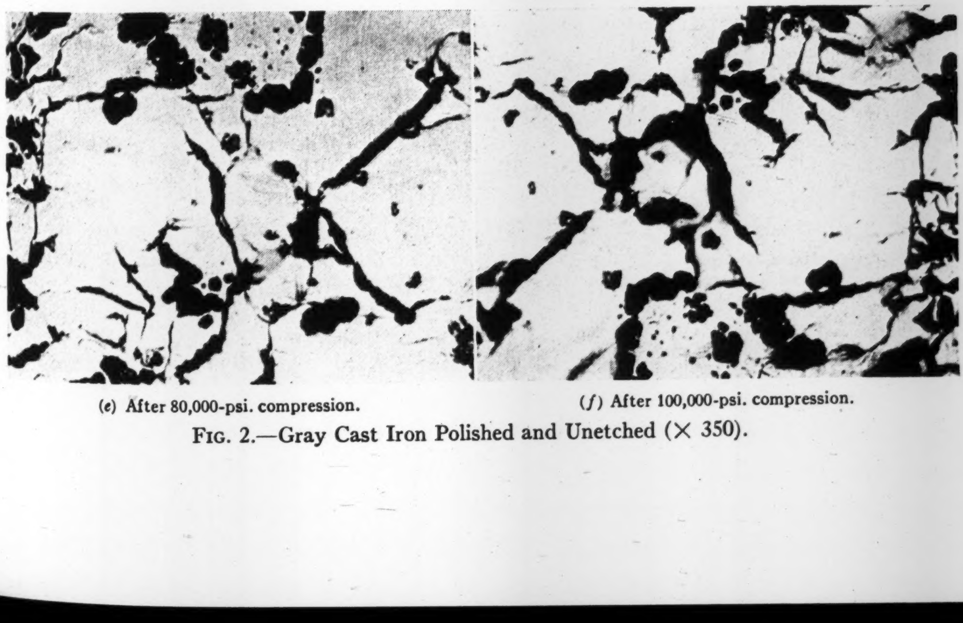
(*a*) Before loading.

(*b*) After 20,000-psi. compression.



(*c*) After 40,000-psi. compression.

(*d*) After 60,000-psi. compression.



(*e*) After 80,000-psi. compression.

(*f*) After 100,000-psi. compression.

FIG. 2.—Gray Cast Iron Polished and Unetched (× 350).

of increasing the height of the specimen was to decrease the load at which final fracture took place. The general trend in all five series of tests is the same.

It is one thing to establish a relationship or a trend and quite another matter to explain it. In a search for a plausible explanation of this apparent violation of the theories of strain hardening, it was first noticed that as the point of fracture was approached the surfaces of the specimen became dull and that after fracture, very small cracks could be observed on these faces. Accordingly, a 1 by 1 by $\frac{1}{2}$-in. specimen was polished for metallographic examination. This specimen was photographed in the unetched condition at a magnification of 350 after 0, 20,000, 40,000, 60,000, 80,000 and 100,000 psi. loadings. (See Fig. 2.) The specimen was oriented on the microscope stage so that the same spot on the specimen was always in the field of view.

By studying these photomicrographs, the progressive development of fine cracks can be observed. No marked change can be observed until after the 60,000 psi. loading when the development of the cracks is clearly visible. This is the point where the hardness after loading begins to fall below the original hardness before loading. From that point on, the development of the cracks is very plain.

From these observations it was concluded that the application of compressige loads greater in intensity than 40,000 psi. produced intercrystalline fissures in gray cast iron, the size of the fissures increasing with the intensity of the compressive load. When tested in the Rockwell hardness tester, using the B scale penetrator ($\frac{1}{16}$-in. ball), the penetrator would slip into one of these intercrystalline fissures and give a reading that would be interpreted as a decrease in hardness.

If this reasoning is sound, then readings made with the Brinell hardness tester should not show such a decrease in hardness because of the larger penetrator (10-mm. ball) which would integrate over a larger area, possibly larger than any intercrystalline fissure. An attempt was made to check this conclusion, but the difficulties attendant upon obtaining a sufficient number of Brinell hardness readings to obtain a reliable mean value forced the abandonment of the attempt, for the time being.

As a final warning, it must be emphasized again that due to the variable nature of the material under investigation, the generalizations and conclusions developed by this investigation can have only the most general application and interpretation. However, the author feels that, at least from the standpoint of students in the testing laboratory, his purpose has been accomplished, and any wider interest or application that may develop from this study is in the nature of an unearned increment.

Acknowledgment:

The author was fortunate to have the assistance of two of his students in this investigation. Harold Horwitz, senior in Civil Engineering, was responsible for the major portion of the routine hardness testing and computation and E. J. McMahon, senior in Chemical Engineering, did all of the metallographic and photographic work.

DISCUSSION

MR. JOHN N. KENYON[1] (*presented in written form*).—I believe that Mr. Peck has made a substantial contribution by calling attention to an apparent exception to the commonly accepted theory of strain hardening, and by showing that the Rockwell hardness machine is a sensitive instrument for determining the progress of fissures in cast iron when stressed beyond a certain limiting value.

Research usually develops interesting leads for further studies. It might therefore be advisable to carry the investigation farther and extend it to cast irons, such as Meehanite, that consist largely of pearlite with finely dispersed free carbon. Perhaps the hardness impressions should be taken on a surface machined from the center of the test specimens in order to get away from the restraining effect at the load-bearing surfaces.

MR. J. T. MACKENZIE.[2]—This paper deals with only one specimen of cast iron, apparently of compressive strength a little over 100,000 psi., so it would be unwise to generalize from this one set of tests, interesting though it is. Cast irons of well over 200,000 psi. are regularly made and the point of damage on these would presumably be much higher than the 60,000 psi. given in the paper.

MR. JOHN SANFORD PECK (*author's closure by letter*).[3] Mr. MacKenzie's remarks are axiomatic and require no answer. In fact the author himself was most careful to emphasize the dangers of generalizing beyond the data. Mr. Kenyon's suggestions for further research were exactly the comments which the author hoped to obtain and if the time factor can only be satisfactorily adjusted, such research will continue.

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YOUNG'S MODULUS OF ELASTICITY AND SOME RELATED PROPERTIES OF GRAPHITIC MATERIALS[1]

By H. A. Schwartz[2] and C. H. Junge[2]

Synopsis

Young's modulus of elasticity, density, resistivity and coercivity of a fairly extensive series of alloys comprising ingot iron, steel, graphitizable steel, white cast iron, malleable and pearlitic malleable cast iron, and gray cast iron were determined and correlated with chemical composition and heat treatment with special emphasis on the combined and graphitic carbon content. Consideration is given to the relative consistency of results of various evaluations of Young's modulus of elasticity.

An extremely close correlation between density and modulus of elasticity is demonstrated, the correlation being closer than that of Young's modulus and graphite content. A metallurgical explanation giving a new demonstration of the relative solubility rate of graphite and cementite is offered. Only a loose correlation of combined and graphitic carbon and the electromagnetic properties is found to exist. Some consideration is given to the effect of geometric form of graphite.

This paper represents a portion of a much larger study long in progress in the Research Department of the National Malleable and Steel Castings Co. which will it is hoped lead to a comprehensive and logical understanding of the physical and engineering properties of the cast graphite-bearing ferrous alloys.

It is plain that in these grossly heterogeneous mixtures, containing graphite and a ferrous alloy, an understanding of the effect of the inclusions in interrupting the continuity of the metallic matrix is of primary importance. This effect can evidently best be observed with respect to properties not greatly affected by the composition of the matrix and greatly different for the matrix and graphite. Young's modulus of elasticity, but insignificantly affected by changes of composition and heat treatment, is evidently well suited to such an investigation and the density almost equally so. It will presently appear that in a restricted field of alloy types an unexpected intimate relation between these variables exists whose effect is the same whether the difference in density is due to graphite or voids.

Resistivity and coercivity, which lend themselves readily to observation, were included but were not found to fall so well into a logical picture. This was not altogether unexpected in view of the unsatisfactory state of our knowledge of the systematic relation between chemical composition, heat treatment, and resistivity of steels, or of the principles underlying the resistivity of heterogeneous systems as an electrical problem.

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[1] The data of this paper are contained in a thesis presented to Case School of Applied Science by Mr. Junge in partial fulfillment of the requirements for the degree of Master of Science in Metallurgical Engineering, June, 1941.

[2] Manager of Research, and Research Metallurgist, respectively, National Malleable and Steel Castings Co., Cleveland, Ohio.

MATERIALS

The materials were selected to furnish a comparatively complete coverage of the ferrous alloy field excluding the use of alloying elements almost entirely. They

TABLE I.—CHEMICAL COMPOSITION AND HEAT TREATMENT.

Material		Treatment	Composition, per cent							
			Total Carbon	Graphitic Carbon	Combined Carbon	Silicon	Manganese	Chromium	Molybdenum	Nickel
Ingot iron	No. 1	2½ hr. 700 C., air cooled	0.012	....	....	trace	0.017	....	....	....
S.A.E. 1045	No. 2	45 min. 825 C., furnace cooled	0.45	....	....	0.35	0.75	....	....	....
	No. 3	45 min. 825 C., water quench; 2 hr. 650 C., air cooled	0.45	....	....	0.35	0.75	....	....	....
Nickel - chromium - molybdenum steel.	No. 4	45 min. 825 C., furnace cooled	0.75	....	....	0.20	0.70	0.75	0.30	1.75
	No. 5	45 min. 825 C., water quench; 2 hr. 650 C., air cooled	0.75	....	....	0.20	0.70	0.75	0.30	1.75
Alloy cast iron	No. 6	None	2.27	1.61	0.66	2.62	1.04	....	0.40	2.87
Meehanite	No. 7	None	3.23	2.35	0.88	1.18	1.10	....	....	....
	No. 8	None	3.21	2.37	0.84	1.22	0.94	....	....	....
Mallix	No. 9	Interrupted malleable anneal	2.30	1.61	0.69	0.99	0.78	....	....	....
	No. 10		2.56	1.68	0.88	0.91	0.75	....	....	....
Heat-treated malleable iron	No. 11	1½ hr. 840 C., oil quench; 1½ hr. 690 C., air cooled	2.54	1.68	0.86	0.91	0.35	....	....	....
	No. 12		2.56	1.67	0.89	0.91	0.35	....	....	....
	No. 13	1½ hr. 840 C., water spray; 6 hr. 720 C., air cooled	2.32	1.55	0.77	0.99	0.78	....	....	....
	No. 14	1½ hr. 840 C., oil quench; 1½ hr. 690 C., air cooled	2.59	1.88	0.71	1.04	....	....	....	....
	No. 15		2.63	1.90	0.73	0.96	0.37	....	....	....
	No. 16		2.54	1.75	0.79	1.06	0.39	....	....	....
	No. 17		2.59	1.79	0.80	1.06	0.39	....	....	....
	No. 18	Same after interrupted malleable anneal	2.53	1.79	0.74	....	....	....	....	....
Malleable iron	No. 19	1½ hr. 840 C., oil quench; 1½ hr. 690 C., air cooled	2.59	2.07	0.52	....	....	....	....	....
	No. 20	45 min. 860 C., air cooled	2.53	....	....	0.91	0.35	....	....	....
	No. 21	Commercial 3 day anneal in Dressler kiln	2.51	2.49	0.02	0.91	0.35	....	....	....
	No. 22		2.53	2.50	0.03	1.06	0.39	....	....	....
	No. 23	Dressler anneal; 45 min. 860 C., air cooled; 3 days 700 C.	2.53	2.49	0.04	0.91	0.35	....	....	....
	No. 24	Dressler anneal, HTM, then Dressler re-anneal	2.58	2.56	0.02	1.04	....	....	....	....
	No. 25	Commercial Dressler anneal	2.57	2.39	0.18	....	....	....	....	....
	No. 26	Graphitized below A_1 point	2.52	2.52	0.00	....	....	....	....	....
	No. 27	Commercial Dressler anneal	2.50	....	....	0.91	0.35	....	....	....
	No. 28[a]		2.63	2.63	0.00	....	....	....	....	....
	No. 29	Prequenched from 825 C., then Dressler anneal	2.62	2.60	0.02	....	....	....	....	....
	No. 30	Prequenched from 950 C., then Dressler anneal	2.64	2.64	0.00	....	....	....	....	....
	No. 31	Commercial Dressler anneal	2.61	2.52	0.09	....	....	....	....	....
White cast iron	No. 32	None	2.64	0.00	2.64	....	....	....	....	....
Graphitizable steel	No. 33	½ hr. 900 C., air cooled below 650 C.; 1 hr. 760 C., cooled to 540 C. in 5 hr., air cooled	1.46	0.89	0.57	....	....	....	0.20	....
	No. 34	Same, then Dressler anneal	1.42	1.06	0.36	....	....	....	0.20	....
	No. 35	½ hr. 900 C., air cooled below 650 C.; 1 hr. 760 C., cooled to 540 C. in 5 hr., air cooled	1.40	0.21	1.19	0.97	0.59	0.48	....	....
	No. 36		1.44	0.63	0.81	1.04	0.62	....	0.19	....
	No. 37		1.45	0.64	0.81	1.02	0.55	....	0.20	....
	No. 38		1.42	0.75	0.67	0.93	0.56	0.14	....	....
	No. 39		1.39	0.78	0.61	0.93	0.56	0.14	....	....
	No. 40		1.03	0.63	0.40	1.25	0.66	....	0.12	....
	No. 41		1.05	0.63	0.42	1.33	0.64	....	0.14	....
	No. 42	2 treatments as above	1.08	0.80	0.28	1.33	0.64	....	0.14	....
	No. 43	Spheroidizing anneal	1.04	0.30	0.74	1.24	0.70	....	0.15	....
	No. 44		1.00	0.58	0.42	....	....	....	....	....
	No. 45	1 hr. 900 C., air cooled to 650 C.; 1 hr. 760 C., furnace cooled to 650 C.	1.03	0.01	1.02	1.32	0.75	0.39	0.15	....

[a] Old-style bar.

include the purest commercially available iron, a plain carbon and an alloy steel, a white cast iron, a fair number of partially and completely graphitized materials produced by various heat treating processes whose significance will be more apparent in the later discussion, a series of the relatively new graphitizable cast steels, and two types of rather high quality gray iron, one being a nickel alloy. We are indebted to the Fulton Foundry Co. of Cleveland and to the research laboratory of the International Nickle Co. for these gray irons. The materials are described as to chemical composition and heat treatment in Table I.

EXPERIMENTAL METHODS

The determination of modulus of elasticity follows the familiar pattern of plotting a stress-strain curve and evaluating its slope. The specimens were 0.505 in. in diameter with an 8-in. gage length. The ends were threaded for screwing into the conventional spherical seated specimen holders. The testing machine was a rather light manually operated Riehle machine originally of 30,000-lb. capacity rebuilt in our laboratory to decrease its capacity and increase its sensitivity in the ratio of 5 to 1. Care was taken in the reconstruction to maintain the center of gravity of the beam system in proper relation to the central knife edge. The machine was calibrated by dead loading and found to be in error by an amount less than is accurately measurable which is of the order of 1 lb., assuming the accuracy of the calibrating weights. These errors are disregarded in our subsequent considerations. Strain was measured by a Ewing extensometer whose scale was calibrated in terms of a single turn of the instrument's micrometer screw. The pitch of that screw was compared with that of an infrequently used Gaertner micrometer screw through the intervention of an Ames dial and no detectable difference was found. Even if there should be unrecognized errors of calibration they persist through the entire investigation and are of minor importance since accurately comparative values are more important for our purposes than absolute precision.

The stress-strain curves plotted from the observations were quite satisfactorily smooth, departing from their trend lines by amounts readily accounted for by the degree of precision with which fractions of extensometer divisions could be interpolated. Their evaluation in terms of modulus of elasticity could be made in two ways. The lines were, by inspection, all nearly straight up to 8000 psi. and the data up to this limit were used in computation. Up to 5000 psi. readings were taken at intervals of 250 psi., then at intervals of 500 psi. to about 20,000 to 30,000 psi., depending upon the material. No corrections for testing machine or extensometer errors was attempted. Either the slope of the best straight line representing the points as computed by least squares from the observational data can be taken to represent the modulus of elasticity throughout or the locus (computable by least squares) can be considered as a quadratic function of stress whose first derivative when stress is zero represents the modulus of elasticity at the origin. The fact that the coefficient of the square of stress is sometimes negative and the slope at the origin highly erratic and often of most unlikely value seems sufficient reason for discarding the latter method of evaluation; however, the data are retained for purposes of record. As a check we measured the slope of the line obtained by joining the ends of the very narrow, hysteresis loop obtained when varying the load between the limits 2000 psi. and 10,000 psi. which yields a value of modulus of elasticity applicable, presumably, near 5000 psi.

The specimens were baked 24 hr. at 230 C. to remove cold work before these observations were made. Density was determined by the Archimedean method correcting for the coefficient of expansion of water, but uncorrected to vacuo. The density is ostensibly in grams per cubic centimeter at 25 C., which is near laboratory temperature, but no correction for the coefficient of thermal expansion of metal over the short intervals by which the real temperature departed from 25 C. from time to time was made.

TABLE II.—PHYSICAL PROPERTIES.

	Young's Modulus of Elasticity, psi. ($\times 10^{-6}$)			Density, g. per cu. cm.	Resistivity, microhms per cu. cm.	Coercivity, oersteds	Brinell Hardness Number
	E^a	E_l	E_h				
No. 1	30.21	28.74	.....	.....	.....	.....	...
No. 2	32.97	29.52	30.72	.....	16.96	.....	...
No. 3	31.83	30.33	31.57	.....	16.06	.....	...
No. 4	33.24	30.80	31.34	.....	26.96	.....	...
No. 5	28.87	29.49	30.70	.....	25.86	.....	...
No. 6	20.52	19.48	20.06	7.3160	77.14	29.21	373
No. 7	20.94	19.18	20.00	7.2917	53.99	9.58	222
No. 8	20.29	19.07	20.05	7.2885	52.70	9.70	208
No. 9	26.61	26.85	26.71	7.4466	38.19	7.87	222
No. 10	27.60	26.08	26.92	7.4211	38.32	7.64	222
No. 11	22.14	22.59	22.72	7.2122	38.69	10.27	182
No. 12	24.75	23.28	23.03	7.2121	38.73	12.89	188
No. 13	27.55	26.54	26.61	7.4362	38.13	12.09	208
No. 14	25.79	22.64	22.99	7.2036	39.26	11.41	171
No. 15	21.70	21.71	23.37	7.2047	38.19	8.44	194
No. 16	25.46	23.51	23.58	7.2072	41.17	12.55	194
No. 17	22.46	22.81	22.86	7.2104	40.96	9.58	194
No. 18	30.12	27.84	26.74	7.3916	37.23	11.41	208
No. 19	25.25	24.27	24.60	7.2621	36.71	9.13	161
No. 20	23.57	22.54	.....	.....	.....	.....	...
No. 21	24.11	24.08	26.29	7.2755	31.23	1.19	104
No. 22	27.19	25.39	25.75	7.2609	34.12	1.16	112
No. 23	22.58	22.14	23.35	7.2521	33.70	7.36	115
No. 24	19.28	19.96	22.64	7.0911	35.52	1.92	106
No. 25	26.21	25.11	25.61	7.2729	34.36	3.26	128
No. 26	25.54	25.04	24.96	7.2162	32.84	2.42	124
No. 27	24.52	24.05	.....	.....	.....	.....	...
No. 28	28.43	26.07	25.46	7.2645	31.04	2.58	106
No. 29	26.01	25.56	26.35	7.2610	30.60	4.67	117
No. 30	27.40	26.08	26.11	7.2628	30.15	3.51	118
No. 31	27.44	25.71	25.17	7.3162	28.84	1.21	109
No. 32	.....	28.11	27.85	.....	49.11	.....	...
No. 33	28.88	27.65	28.54	7.5603	38.11	9.47	208
No. 34	32.37	26.52	26.15	7.3851	39.27	4.61	161
No. 35	39.32	32.10	30.00	7.6820	39.66	14.95	248
No. 36	29.93	29.46	28.92	7.6160	37.39	15.18	215
No. 37	30.32	28.98	29.49	7.6110	37.55	15.06	208
No. 38	28.51	27.57	29.77	7.6000	35.41	8.90	222
No. 39	27.37	27.70	29.72	7.5992	35.86	9.25	222
No. 40	36.14	31.44	28.74	7.6217	37.61	10.95	208
No. 41	26.78	28.22	29.14	7.5954	39.16	8.67	194
No. 42	29.23	27.18	27.49	7.5229	38.90	8.10	176
No. 43	30.14	29.26	29.49	7.6705	.....	13.01	215
No. 44	27.13	27.69	29.09	7.6229	37.80	9.93	188
No. 45	33.20	30.30	30.09	7.7174	41.42	14.49	257

[a] Determined from slope of quadratic at origin; rejected.

Resistivity was measured on the Young's modulus test specimens; a second strain relief might have been desirable but was inadvertantly neglected. The measurements were made on a Leeds & Northrup Kelvin Bridge which was assumed correct without further confirmation. Coercivity was determined for us by Thomas R. Graham and William W. Culbertson, Instructors in Metallurgy at Case School of Applied Science to whom our thanks are due. Graham and Culbertson used a coercimeter, built at Case, of the type employed by Clayton in his investigations at the Missouri School of Mines. We are unfamiliar with any further details.

The physical properties are summarized in Table II.

The total and graphitic carbon recorded in Table I were determined for us by G. M. Guiler in this laboratory, on specimens consisting of solid pieces, not chips. Duplicates on total carbon very rarely differed by 0.02 per cent and on graphite by 0.03 per cent; usually exact checks were obtained. It is a pleasure to record our thanks for this cooperation.

When composition with respect to other elements is referred to, the information is based on works analyses or specifications and the numerical data have only a descriptive significance. No important quantitative conclusions are based on such figures.

DISCUSSION OF DATA

Most of our observations can best be summarized by the usual statistical methods.

Our first step is a comparison of the two values of Young's modulus of elasticity, E_l determined from the best straight line, and E_h determined from the hysteresis loop. The correlation coefficient of the two moduli calculated from the data for all the specimens was found to be 0.952. The lines of regression were found to be

$$E_h = 2.0568 + 0.9373\ E_l \text{ and}$$

$$E_l = -\ 0.8593 + 1.1062\ E_h$$

The line of best fit (minimizing the residuals perpendicular to this line) is

$$E_h = 2.1350 + 0.9343\ E_l$$

Throughout the discussion the unit of stress is 1,000,000 psi. From the above equation $E_h > E_l$ if $E_l < 32$ as it always is.

In comparing carbon content and modulus of elasticity one is at first glance tempted to regard graphite as the cause and the modulus as the effect and also to consider that the graphite content is the more accurately known variable. A line of regression of Young's modulus on graphite is indicated. If one did not choose to disregard any effect of combined carbon as a variable then a plane of regression of the modulus on the graphite - combined carbon plane may be computed. Taking the data for graphitic systems in which there has been no recombination of carbon under heat treatment and excluding gray iron—subjects to be referred to later—the plane of regression was found to be

$$E_l = 29.8495 - 1.8019\ G + 0.2265\ C$$

where G = graphite, and
C = combined carbon in per cent.

The contribution made to Young's modulus by combined carbon is evidently small and on the verge of being problematical. The use of lines of regression thus seems justified, omitting combined carbon as a variable. The equations of these lines are found to be

$$E_l = 30.2034 - 1.9682\ G, \text{ and}$$

$$G = 12.1771 - 0.3913\ E_l$$

The "line of best fit" has the equation

$$E_l = 30.9346 - 2.4354\ G$$

and the correlation coefficient of G and E_l is -0.888. The individual points fall well within the tolerances required for a homogeneous system. Be it noted, however, that an attempt to apply this equation to gray iron results in an average value of over 26 on three specimens where the observed value was but little over 19, thus indicating that the relationship is a function of the geometric form of the graphite.

Similar calculations based on E_h give the following equations:

$$E_h = 30.6489 - 2.0340\ G - 0.2129\ C$$

$$E_h = 30.2798 - 1.8566\ G$$

$$G = 15.2997 - 0.5017\ E_h$$

$$E_h = 30.4474 - 1.9637\ G$$

The correlation coefficient is -0.965 showing that E_h is somewhat more consistently related to graphite than is E_l.

Again using the data for the same materials and computing a relation of density and graphite we have the lines of regression

$$\rho = 7.7247 - 0.1798\ G$$

$$G = 42.5513 - 5.5065\rho$$

a line of best fit

$$\rho = 7.7248 - 0.1799\ G$$

and a correlation coefficient of -0.995, and a plane of regression

$$\rho = 7.7284 - 0.1811\ G - 0.0039\ C$$

The application of the above equation to the three gray iron specimens gives an average calculated density of 7.3432 as against an observed value of 7.2987. Although the discrepancy is not so glaring as in Young's modulus of elasticity, the individual readings fall far out of the permissible tolerance from the line of regression and it is plain that the effect of graphite on density is quantitatively different in gray iron than in materials containing temper carbon in the direction that flaky graphite is apparently a less dense aggregate.

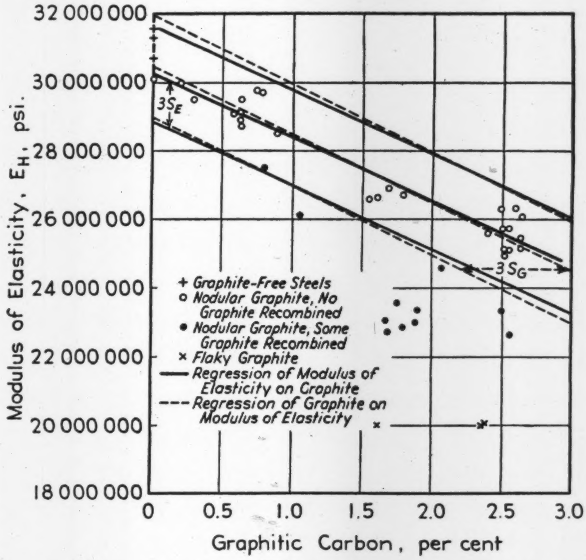

FIG. 1.—Relation of Modulus of Elasticity and Graphite Content.

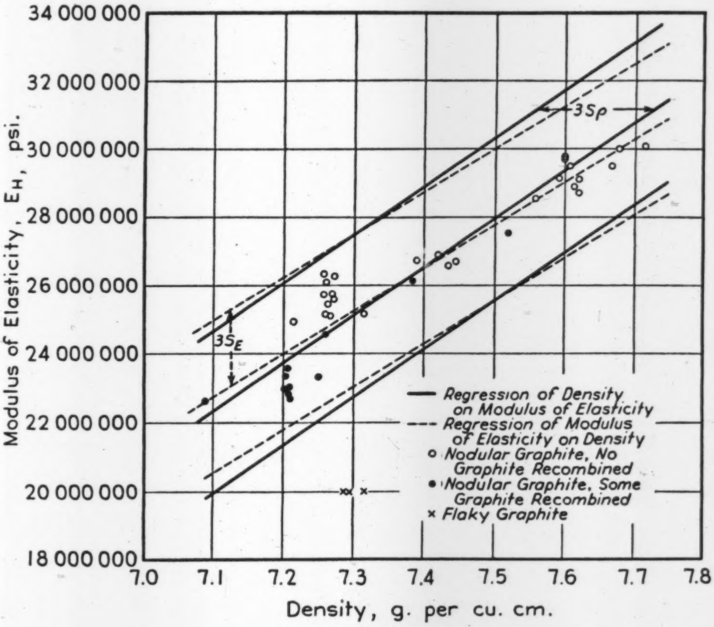

FIG. 2.—Relation of Modulus of Elasticity and Density.

Figures 1 and 2 show the observed values of the modulus of elasticity for observed values of graphite content and density, respectively. The agreement with the calculated values may be judged by comparison with the lines of regression and the "scatter bands" which are included in the figures.

The densities of materials in which recombination of carbon has been brought about is far less than that expressed by the above equations. Passing through calculations based on specific volume, one may calculate the apparent densities of ferrite, graphite, and cementite, reaching the values 7.7304, 2.2082, and 7.3312, respectively. The data are in fair, although not precise, agreement with direct determinations. Considering the long extrapolation to the density of pure graphite and pure cementite, perhaps the agreement is all that can be expected. The fact that all the values are low may represent the difficulty in obtaining a perfectly sound specimen in cast form.

Certain experiments, not here reported, demonstrated that when ferritic materials are heated to recombine carbon a void is left, but not when pearlitic materials are so treated. Prompted by this the density data of ferritic alloys reheated to recombine carbon were studied on the assumption that the densities of ferrite, graphite, and cementite were those calculated from the preceding equations and that their reduced density represented voids produced by the solution of the recombined amount of graphite. The apparent density of graphite in materials when recombination has occurred computed as before is 1.57 as compared with a probable value of 2.2082. The graphite is thus accompanied by a void whose volume is about 0.41 that of the graphite. This number is of course specific to the amount of carbon recombined which averages 0.86 per cent in material whose graphite averages 1.79. The ratio is 0.48 as against 0.41 which agrees well with an assumption that the volume of void left by recombination of a given amount of carbon approximates the equivalent volume of graphite. We need follow this subject no further for the purpose of this paper.

Having seen that close correlation exists between modulus of elasticity and graphite and between density and graphite, both in the absence of recombination of graphitic to combined carbon, no formal demonstration is needed that modulus of elasticity and density are closely related. It is another matter to determine whether this correlation exists when this density is affected by voids. Calculation using all graphitic specimens except gray iron gives the following lines of regression:

$$E_h = 12.5436\rho - 66.3113$$

$$\rho = 0.071548\, E_h + 5.5017$$

$$E_l = 13.8106\rho - 76.0059$$

$$\rho = 0.059669\, E_l + 5.8347$$

The correlation coefficients are 0.947 and 0.908, respectively, and the two lines of best fit:

$$E_h = 13.9685\, \rho - 76.8359$$

$$E_l = 16.7465\, \rho - 97.6902$$

All the observations fall into the tolerance limits for a homogeneous system. It is apparent that density and modulus of elasticity remain closely related even when the former is affected by voids.

The electromagnetic properties do not deserve much discussion. We have been unable to find in the literature any entirely satisfactory treatment of the resistivity of a heterogeneous system or of the effect of alloying elements, especially in complicated systems, on the resistivity of iron. Using Benedick's[3] conclusions in the latter field to adjust all resistivities to the composition 1 per cent silicon, 0.4 per cent manganese it is possible to set up the equation

$$R = 32.396 + 0.8577\,G + 3.6285\,C$$

for the resistivity of our graphitized alloys excluding gray iron. Using Norbury's[3] data on the effect of alloys on resistivity and the ideas advanced in "Science of Metals" by Jeffries and Archer for the conductivity of a heterogeneous aggregate, one may calculate a plane of regression of the product of specific volume and specific conductivity on graphite and carbon. For an alloy containing 1.0 per cent silicon and 0.35 per cent manganese we obtain

$$V\lambda = 0.00448125 - 0.0000588\,G - 0.0008356\,C$$

where V and λ are the specific volume and conductivity, respectively. Values of $V\lambda$ calculated from this equation for ferrite, cementite and graphite bear no reasonable relation to the constants so far as they are known. Obviously our basic knowledge is insufficient and the equation can have but empiric significance over a restricted range of composition.

In the evaluation of the coercivity data, the properties of similar materials—malleable, pearlitic malleable, graphitizable steel, and steel—were averaged for each group and a plane of regression of coercivity on graphite and combined carbon computed as:

$$F = 6.4506 - 1.7580\,G + 8.8579\,C$$

Although the plane passed satisfactorily near the four individual points, extrapolation to pure iron yields an entirely impossible result. Also there is a considerable scatter in the coercivity of the materials in a single group due perhaps to the effect of grain size. The equation is offered as empirical only.

CONCLUSIONS

1. Young's modulus of elasticity may be satisfactorily measured either by fitting the best straight line to a stress-strain diagram or by observing the stress-strain relation on repeated loading and unloading between the limits 2000 psi. and 10,000 psi.

2. The latter method is perhaps preferable both on account of ease of execution and consistency of result.

3. The results by the latter method will be consistently somewhat greater than by the former.

4. In the absence of recombination of graphite in the heat treating process, modulus of elasticity for materials having all their graphite as temper carbon, and graphite content are linearly related.

5. In all materials containing only the temper carbon form of graphite, modulus of elasticity and density have a close linear relationship.

6. Graphite in the flaky form decreases modulus of elasticity much more than an equal amount of temper carbon.

7. These principles may perhaps form the basis for quantitatively expressing graphite form.

8. The re-solution of carbon from cementite is enormously faster than from graphite.

9. Empirical equations are given correlating resistivity and coercivity with carbon content which probably have little theoretical significance.

10. When graphite exists only as temper carbon, that is, when the metallic matrix is interrupted only by approximately spherical voids and/or inclusions, modulus of elasticity is directly proportional to the amount of metallic phase in a unit volume.

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[3] H. E. Cleaves and J. G. Thompson, "The Metal-Iron," p. 188, McGraw-Hill Book Co., Inc., New York, N. Y. (1935).

DISCUSSION

Mr. H. F. Moore.[1]—This is an unusual paper in that the authors, in making determinations of modulus of elasticity, have taken care to use specimens with relatively long gage lengths carefully measured, to use a carefully calibrated strainometer, and a carefully calibrated testing machine. They have then used statistical methods in determining the value of modulus of elasticity from their data. A good many values of modulus of elasticity have been reported (some of them, I fear by myself) in which sufficient care about such details has not been taken. If elastic constants are to be accurately determined, methods like those described in this paper, or others involving as great care and sensitivity, are necessary.

Mr. J. S. Vanick.[2]—The authors point out that cast irons with flake graphite do not regiment themselves into these diagrams. We would probably be disappointed if they did, because of the extreme importance of flake distribution as well as graphite quantity in the case of gray cast iron. Coming back to the malleable type of iron, the authors seem to suggest the possibility that even when the graphite was in a nodular form, if it had been formed at a higher temperature, the product might be less dense and therefore lower in elastic modulus. I should like to have them confirm or restate the point as to whether the higher temperature anneal of malleable would lead to that kind of result.

Messrs. H. A. Schwartz[3] **and C. H. Junge**[3] (*authors' closure, by letter*).—The authors are very appreciative of Mr. Moore's words of commendation in regard to their paper.

We had not intended to suggest what Mr. Vanick apparently understood us to say, namely, that there was any difference between the effect of a given amount of graphite formed at high and at low temperatures. The point which we were making and which has apparently not been clear is that the density and Young's modulus of a material produced from completely graphitized malleable iron by reheating above the critical point are lower than these same properties of a material in which the combined carbon has not been reformed from preexisting graphite but remains as the result of an incomplete graphitization. The same composition with respect to graphite and combined carbon is assumed in both cases.

We have said furthermore that if a material produced by an arrested anneal is reheated above the critical point and again cooled at a rate appropriate for the preservation of the correct proportions of graphitic and combined carbon, then the density and modulus of elasticity will return to the value which they had before the reheat treatment was undertaken.

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Metallurgist, The International Nickel Co., Inc., Bayonne, N. J.

[3] Manager of Research, and Research Metallurgist, respectively, National Malleable and Steel Castings Co., Cleveland, Ohio.

THE TENSILE PROPERTIES OF SOME COPPER ALLOYS

By Cyril Stanley Smith[1] and R. W. Van Wagner[1]

Synopsis

Tension tests were performed on copper and twelve copper alloys in the form of commercially fabricated rods, using an extensometer sensitive to 0.0001 per cent. Typical stress-strain curves are reproduced, together with curves showing offset and permanent set on a large scale, both before and after over-straining in tension. Over-strain decreases the stress first producing measurable offset in annealed alloys, but increases the stress needed to produce an equivalent set. Heat-treated beryllium copper follows Hooke's law to nearly 100,000 psi. if appropriate corrections for true stress and strain are made.

In 1940 one of the authors described[2] a technique permitting stress and strain measurements to be obtained with high accuracy, and gave the results of some tests on a special material. The present paper records the results of some tests of equal sensitivity made on samples of commercially-produced copper alloys.

All the materials tested were in the form of $\frac{3}{4}$-in. diameter rods, fabricated in one of the three Naugatuck Valley mills of the American Brass Co. In each case they were produced by normal manufacturing procedure, to meet certain commercial specifications. Usually, the temper of the rods was that known commercially as "hard." The actual ready-to-finish grain sizes and the amounts of final reduction by drawing are listed, with analyses, in Table I. The tension tests were made on threaded-end specimens of 0.505-in. diameter in the gage length. It is obvious that the properties of the specimen will be somewhat different from those of the full section of the rod; also, that the tests do not represent other tempers or other sizes of the alloys concerned. Additional samples of the hard rod were annealed in the laboratory and tested in this condition.

Method of Test

The method of gripping the test specimens to obtain axial loading, the extensometer (sensitive to 0.0001 per cent on a 2-in. gage length), and other details are given in an earlier paper.[2] Most tests were made at a temperature of 32 ± 0.1 C. in a thermostatically controlled box. Usually, stress and strain readings were taken up to 0.5 per cent total strain (the customary yield point for copper alloys), then the load was released and reapplied to fracture, extensometer readings being taken to about 1 per cent strain.

In the permanent set tests, the load was applied quickly, held 5 sec., and released quickly. Substantially adiabatic conditions were thereby obtained and thermal changes at zero stress were small enough to be dissipated in the two minutes allowed before reading the extensometer. Successive applications of increasing loads were made until a permanent set of 0.100 per cent had been obtained. The specimen was realigned in the holders, allowed to rest about 3 hr. without stress, and then again stressed. The effect of prior tensile strain was thus determined on the stresses producing stated permanent set or offsets, that is, on the "elastic limit" and "proportional limit."

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[1] Research Metallurgist, and Laboratory Assistant, respectively, American Brass Co., Waterbury, Conn.

[2] C. S. Smith, "Proportional Limit Tests on Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 864 (1940).

TABLE I.—COMPOSITION AND TREATMENT OF MATERIAL TESTED.

Alloy	Material	Ready-to-Finish Grain Size	Final Reduction of Area, per cent	Final Grain Size[a]	Composition, per cent						
					Copper	Zinc	Tin	Nickel	Iron	Lead	Other Elements
2956	Tough pitch copper...	0.075	36	0.060	99.950	....	....	....	0.002	....	O 0.043, S 0.002
2957	Oxygen free copper...	0.135	36	0.070	99.996	....	....	....	0.002	....	S 0.002
2948	85-15 brass...........	0.050	3.5	0.125	85.74	14.23	....	....	0.02	0.01	
3032	65-35 brass...........	0.135	33	0.070	63.54	36.42	....	....	0.01	0.03	
3026	Tobin bronze.........	0.030	8	0.032	60.01	39.19	0.76	....	0.01	0.03	
2952	Phosphor bronze A...	0.025	27	0.040	95.35	....	4.28	....	0.02	0.003	P 0.29
2950	Phosphor bronze C...	0.110	30	0.070	91.69	....	8.09	....	0.01	0.01	P 0.11
2951	Phosphor bronze D...	0.120	36	0.090	89.76	0.10	9.96	....	0.02	0.01	P 0.12
3421	Cupronickel..........	0.030	15	....	69.23	0.00	....	30.48	0.07	....	Mn 0.22, Mg 0.03
3420	Ambrac..............	0.060	15	0.060	74.19	5.26	....	20.22	0.08	....	Mn 0.25, Mg 0.06
2901	Everdur 1010.........	0.045	36	0.060	95.75	0.15	0.02	....	0.10	....	Mn 0.95, Si 2.81
2955	Everdur 1015 (H).....	0.14	36	0.130	98.07	....	....	....	0.09	....	Mn 0.30, Si 1.48
2954	Everdur 1015 (XH)...	0.40	64	0.140	97.81	....	....	....	0.04	....	Mn 0.40, Si 1.49
3060	Chromium copper.....	....	84	....	98.96	....	....	....	0.10	....	Si 0.08, Cr 0.93
2992	Beryllium copper (H).	0.075	15	....	97.54	....	....	0.22	0.11	....	Be 2.16
2993	Beryllium copper (A).	....	....	0.055	97.46	....	....	0.21	0.15	....	Be 2.20

[a] Annealed samples only.

RESULTS

The results are tabulated in Tables I and II and summarized graphically in Figs. 1 to 16 and 18 to 29. These figures show for each alloy: (1) a stress-strain loop up to 0.5 per cent extension and release of load, (2A) a part of the same data plotted as offset or deviation from the initial modulus on a large scale, (3) permanent set, and (4A) the same permanent set data plotted on a larger scale. These tests relate to virgin drawn material as received or after annealing. In addition, curves 2B show offset for the same specimen after stressing to 0.5 per cent total strain and release as in curve 1, and curves 4B show the permanent set on restressing after straining to 0.1 per cent set in obtaining curves 3 and 4A. Curves 2A, 2B, 4A, and 4B are plotted to a scale 25 times that of curves 1 and 3 in order to utilize the full sensitivity of the extensometer employed. The figures recorded in Table II are generally based on two determinations and may not exactly agree with the particular curves shown. There are no permanent set curves for the annealed materials.

DISCUSSION

All the annealed alloys have stress-strain curves that are initially straight followed by a well-defined breaking away from proportionality. In some cases they have a definite "drop-of-the-beam" yield point (Figs. 7 and 10),

TABLE II.

Alloy	Material	Condition[a]	Modulus of Elasticity, E, psi., millions	Stress, psi.								Yield Strength (0.5% Strain), psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent
				0.0002% Offset		0.002% Offset		0.0002% Permanent Set		0.002% Permanent Set					
				Initial	Strained[b]	Initial	Strained[b]	Initial	Strained[c]	Initial	Strained[c]				
No. 2956......	Tough pitch copper	H	16.95	8 300	13 400	12 850	21 500	5 000	21 000	13 500	34 800	46 000	46 900	14.8	58.5
		A	15.82	1 150	3 300	1 900	4 720	....	....	....	....	5 500	31 500	53.0	71.4
No. 2957......	Oxygen free copper	H	17.54	8 300	15 000	15 400	24 800	5 500	21 000	14 200	38 200	47 000	47 500	20.5	86.4
		A	16.20	1 400	3 300	1 950	4 500	....	....	....	....	5 000	30 900	60.0	92.1
No. 2948......	85/15 brass	A	17.47	6 450	3 300	7 130	4 900	....	....	....	....	8 600	37 900	58.5	86.6
No. 3032......	65/35 brass	H	16.64	20 200	15 500	26 100	22 500	25 000	36 000	34 200	....	56 500	68 800	20.5	61.5
		A	15.38	11 500	6 600	12 700	8 900	....	....	....	....	12 300	45 400	68.5	77.4
No. 3026......	Tobin bronze	H	14.23	19 300	9 000	22 300	15 000	20 000	27 200	22 500	29 700	32 300	62 000	46.8	62.0
No. 2952......	Phosphor bronze A	H	16.81	30 000	34 500	35 500	46 500	33 500	46 000	39 800	60 200	60 400	71 800	27.6	72.7
		A	17.52	17 400	12 500	18 700	15 800	....	....	....	....	18 700	49 400	73.0	82.1
No. 2950......	Phosphor bronze C	H	14.98	27 500	32 500	34 400	46 000	30 500	40 000	37 200	64 000	60 900	82 800	32.6	72.8
		A	16.78	16 500	14 200	19 300	17 200	....	....	....	....	20 700	56 850	83.0	81.5
No. 2951......	Phosphor bronze D	RA(C)	13.94	38 500	44 300	45 500	52 600	15 800	60 800	44 100	81 000	65 600	99 700	26.6	57.9
		A	16.18	14 900	13 000	21 300	17 900	....	....	....	....	23 100	63 800	75.5	73.2
No. 3421......	Cupronickel	H	21.65	8 000	....	14 500	....	....	....	....	....	63 800	67 300	22.8	70.7
		RA	22.08	31 000	....	36 700	....	....	....	....	....	53 700	65 800	30.5	72.1
No. 3420......	Ambrac	H	20.55	9 300	....	15 500	....	10 100	16 200	24 700	50 800	58 100	61 200	24.8	76.5
		RA	20.77	25 900	....	31 200	....	28 400	37 700	33 500	45 900	45 600	58 300	32.3	77.9
		A	20.25	8 000	....	11 500	....	....	....	....	....	14 350	49 000	50.5	80.4
No. 2901......	Everdur 1010	H	14.64	14 100	15 000	20 000	21 500	30 000	27 000	46 200	63 000	63 200	106 400	15.4	56.2
		A	18.67	10 100	5 100	12 050	7 400	....	....	....	....	15 500	60 800	65.5	76.4
No. 2955......	Everdur 1015	H	16.44	9 800	12 300	15 100	19 500	9 500	17 000	17 500	47 000	53 700	58 800	22.0	81.8
		A	17.90	4 600	3 600	5 400	5 200	....	....	....	....	7 600	37 400	62.0	90.2
No. 2954......	Everdur 1015	XH	15.85	8 900	11 500	14 500	20 000	10 000	22 500	18 200	56 200	62 100	84 600	16.8	75.5
		A	18.00	4 600	3 500	5 150	5 500	....	....	....	....	7 600	38 300	65.0	92.1
No. 3060......	Chromium copper	HT	19.96	18 500	17 000	35 000	35 500	....	....	....	....	74 100	81 300	20.5	55.0
		AT	21.21	19 000	24 000	26 000	27 000	....	....	....	....	46 500	65 300	24.5	60.5
No. 2992......	Beryllium copper	H	16.86	45 300	...	56 600	....	52 500	55 000	63 000	94 000	79 700	110 000	10.7	41.6
		HT	18.49	93 500	....	106 000	....	101 000	165 000	117 500	174 000	91 900	213 000	2.8	3.5
No. 2993......	Beryllium copper	A	17.55	12 000	....	15 500	....	....	....	....	....	29 000	73 300	47.7	56.6
		AT	19.12	59 500	61 000	74 100	93 000	52 000	115 000	71 000	....	94 100	190 800	3.7	6.0

[a] H = Hard drawn. See Table I.
A = Drawn as H; annealed to grain size shown in Table I.
RA = Relief annealed 2 hr. at 450 C.
RA(C) = Commercial relief anneal.
HT = Precipitation heat treatment on hard drawn material. For chromium copper this was 3 hr. at 450 C.; for beryllium copper, 3 hr. at 300 C. Prior to drawing, these materials had been quenched from 975 C. and 825 C., respectively.
AT = Precipitation heat treatment on quenched material. For chromium copper this was 3 hr. at 500 C. following quenching from 1000 C.; for beryllium copper, 3 hr. at 300 C. following quenching from 825 C.

[b] Stress for stated offset after prestraining to 0.5 per cent extension. See Table III for resulting permanent set.

[c] Stress for stated permanent set after prestressing to produce a permanent set of 0.10 per cent.

although the decrease in load is never as great as with mild steel. The cold-worked alloys invariably show no sharp break in the curve and the point of departure from proportionality is sometimes hard to determine. Within the accuracy of measurement, however, there is invariably a straight portion of measurable, sometimes considerable, length. The stress producing 0.0002 per cent offset is frequently far below the usual yield strength, sometimes below the stresses at which the alloys are regularly and safely being used in service. A comparison of two classes of high-strength materials, the tin bronzes and silicon bronzes, discloses great differences in stress-strain be-

Key to numbers of individual curves in Figs. 1 to 15 and 18 to 24.

Curve 1—Stress-strain curve up to 0.5 per cent extension and release of load. Small scale.
Curve 2A—First part of data of curve 1, plotted as offset from assumed modulus. Large scale.
Curve 2B—Offset from assumed modulus, specimen restressed after prestraining as in curve 1.
Curve 3—Permanent set produced by application of progressively increasing loads. Small scale.
Curve 4A—Permanent set data of curve 3, replotted on large scale.
Curve 4B—Permanent set obtained on second test after previous straining to 0.1 per cent set for curve 3.

Note.—In the offset curves, 2A and 2B, the proportionality range is straight but not necessarily vertical unless the assumed modulus was the actual one indicated.

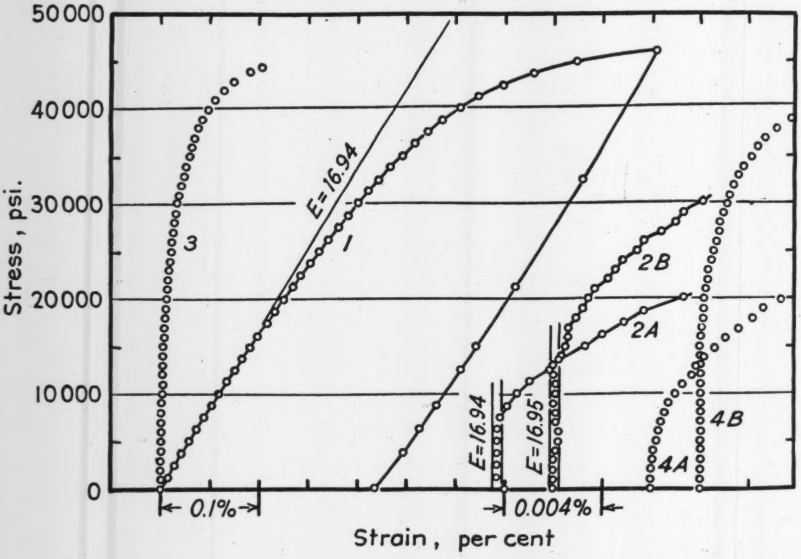

FIG. 1.—Stress-Strain Behavior of Tough Pitch Copper (2956, Cold Drawn).

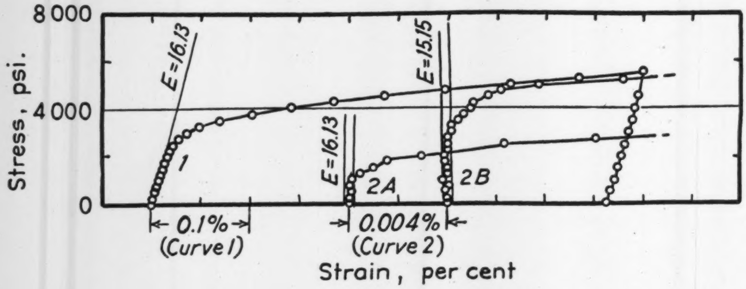

FIG. 2.—Stress-Strain Behavior of Annealed Copper.

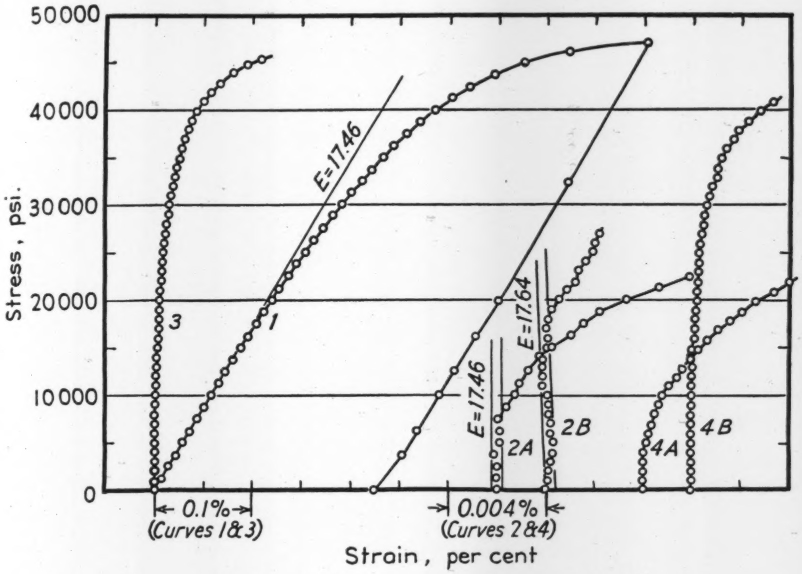

FIG. 3.—Stress-Strain Behavior of Oxygen-Free Copper (2957, Cold Drawn).

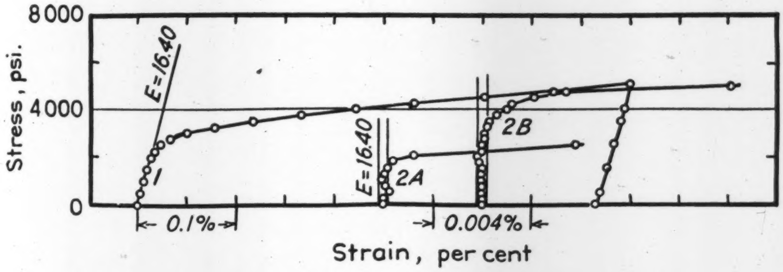

FIG. 4.—Stress-Strain Behavior of Annealed Oxygen-Free Copper.

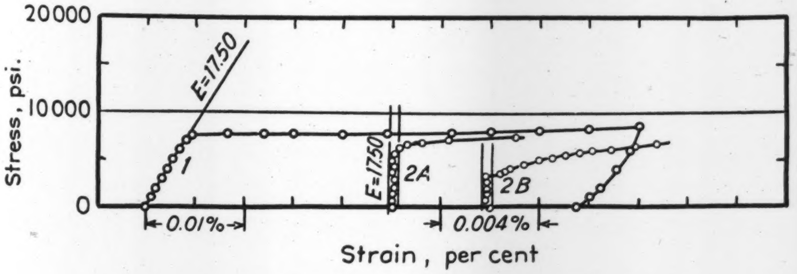

FIG. 5.—Stress-Strain Behavior of Annealed Red Brass (2948).

although the decrease in load is never as great as with mild steel. The cold-worked alloys invariably show no sharp break in the curve and the point of departure from proportionality is sometimes hard to determine. Within the accuracy of measurement, however, there is invariably a straight portion of measurable, sometimes considerable, length. The stress producing 0.0002 per cent offset is frequently far below the usual yield strength, sometimes below the stresses at which the alloys are regularly and safely being used in service. A comparison of two classes of high-strength materials, the tin bronzes and silicon bronzes, discloses great differences in stress-strain be-

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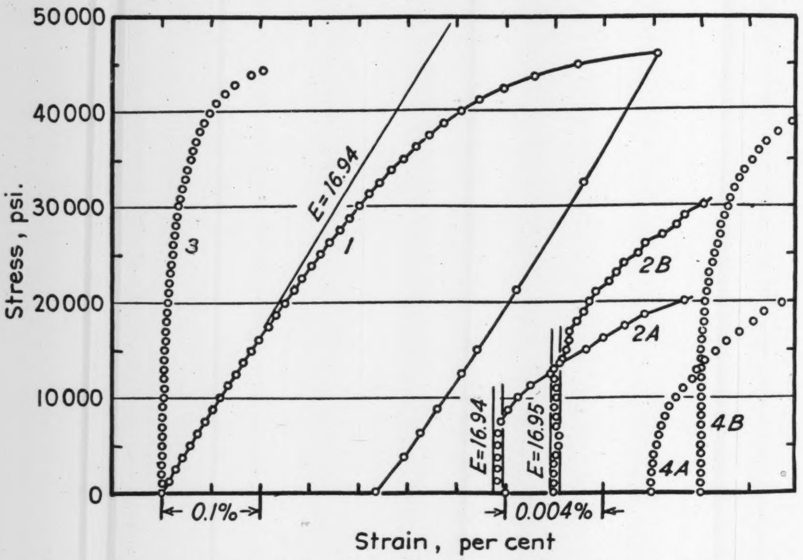

FIG. 1.—Stress-Strain Behavior of Tough Pitch Copper (2956, Cold Drawn).

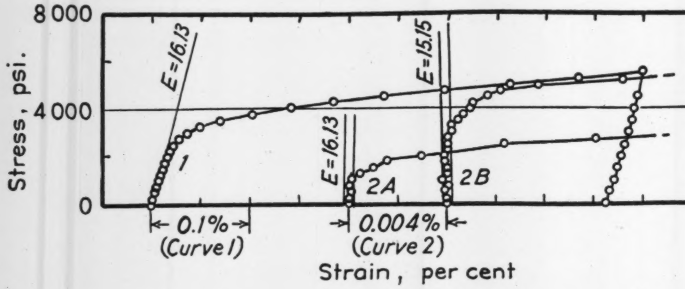

FIG. 2.—Stress-Strain Behavior of Annealed Copper.

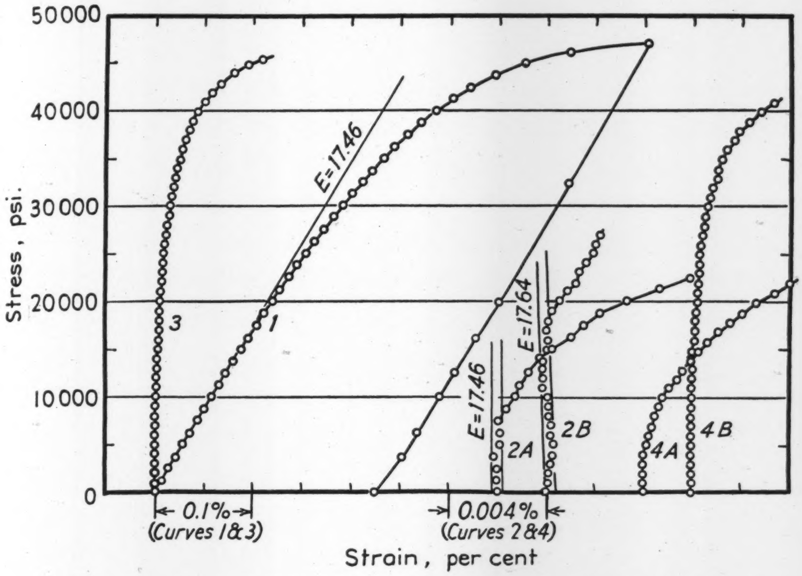

FIG. 3.—Stress-Strain Behavior of Oxygen-Free Copper (2957, Cold Drawn).

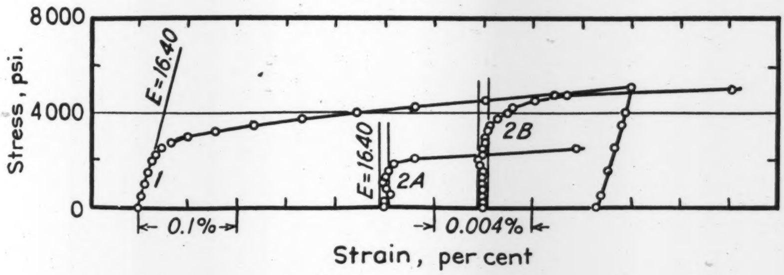

FIG. 4.—Stress-Strain Behavior of Annealed Oxygen-Free Copper.

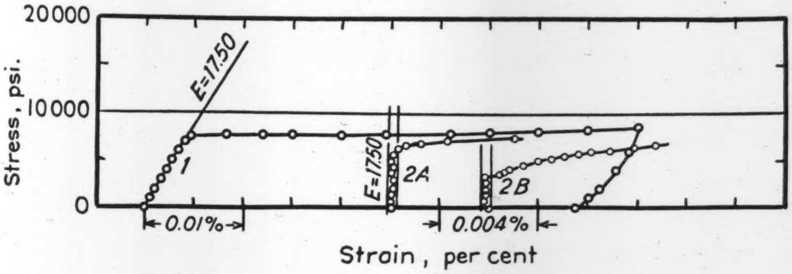

FIG. 5.—Stress-Strain Behavior of Annealed Red Brass (2948).

havior. The cold-drawn 10 per cent phosphor bronze D (Fig. 13) follows Hooke's law up to nearly 40,000 psi., while Everdur (Fig. 18) has a low proportional limit and a stress-strain curve that is straight only up to 14,000 psi. These two materials, however, have roughly equal values for yield strength (0.5 per cent), tensile strength, and endurance limit. The stresses producing 0.002 per cent permanent set are also not much different, though lesser sets vary greatly. It is obvious that deviation from proportionality is irrelevant to service, and, except in the case of elastic measuring members, is of no particular disadvantage. Indeed, Gillett[3] and others have suggested that some degree of disproportionality is a desirable factor in permitting redistribution of stresses in service.

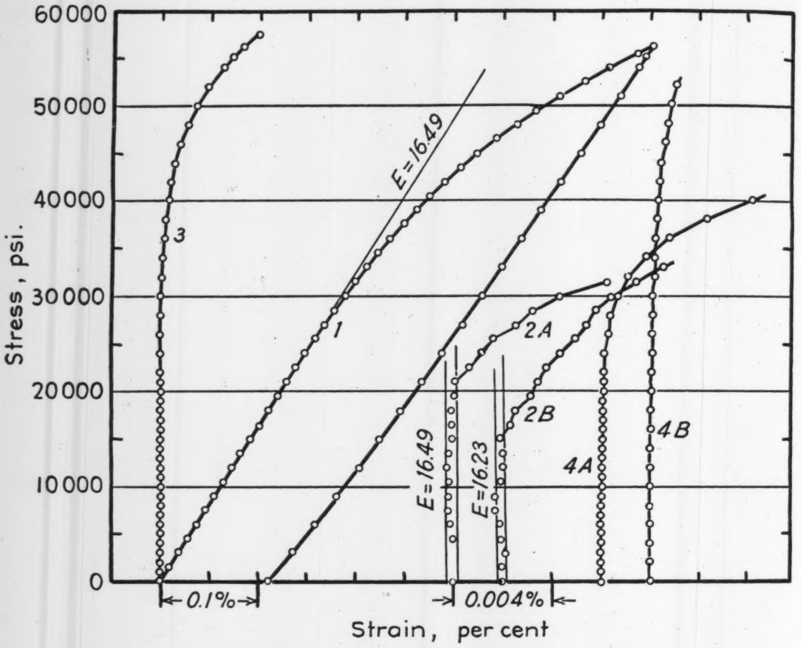

FIG. 6.—Stress-Strain Behavior of 65-35 Brass (3032, Cold Drawn).

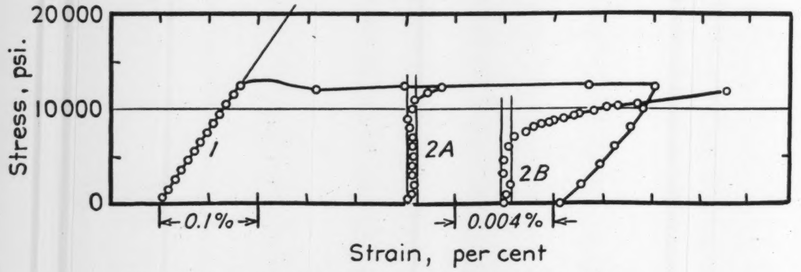

FIG. 7.—Stress-Strain Behavior of Annealed 65-35 Brass.

With most alloys in the cold-drawn condition, the stress producing first

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[3] H. W. Gillett, "The Limited Significance of the Ductility Features of the Tension Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 551 (1940).

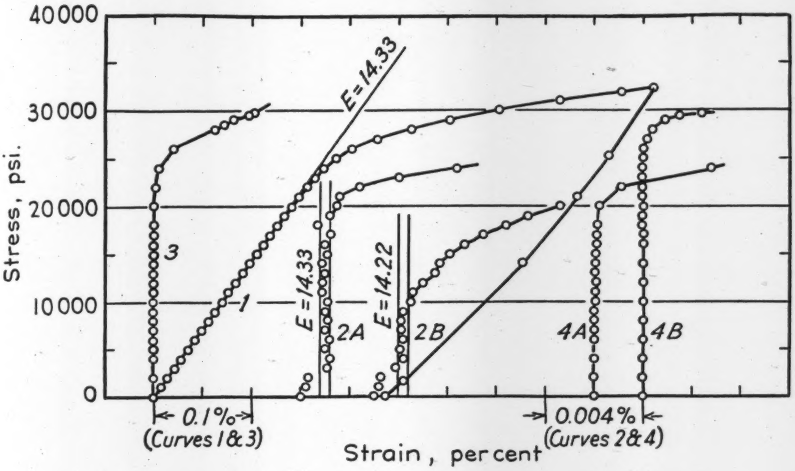

FIG. 8.—Stress-Strain Behavior of Tobin Bronze (3026, Annealed).

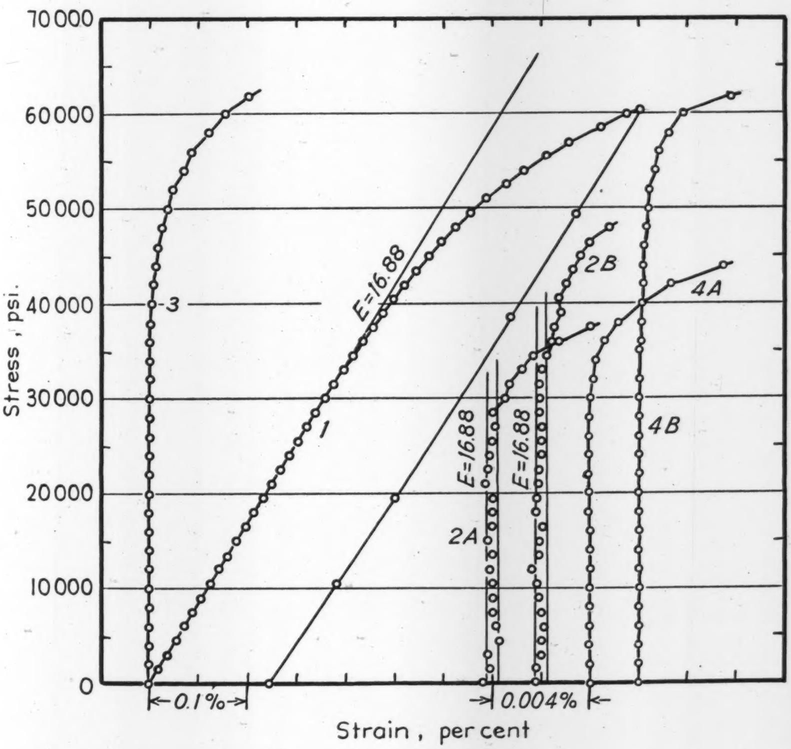

FIG. 9.—Stress-Strain Behavior of Phosphor Bronze A (2952, Cold Drawn).

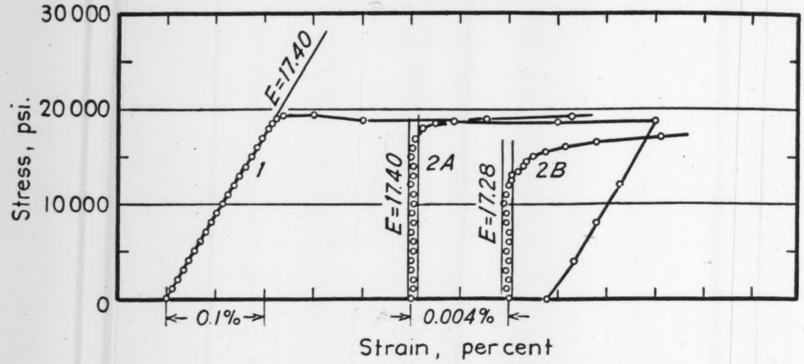

Fig. 10.—Stress-Strain Behavior of Annealed Phosphor Bronze A.

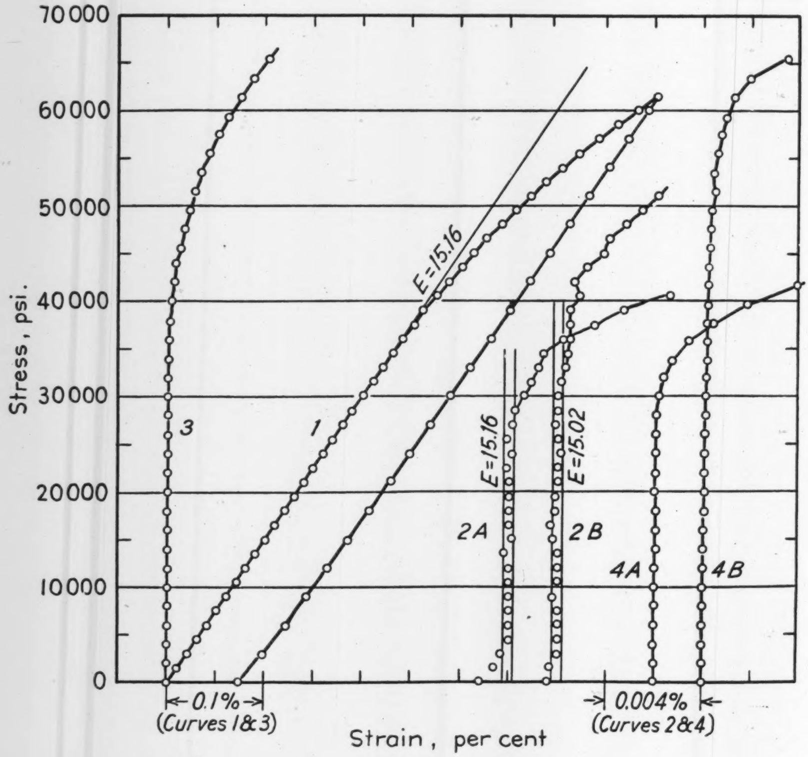

Fig. 11.—Stress-Strain Behavior of Phosphor Bronze C, (2950, Cold Drawn).

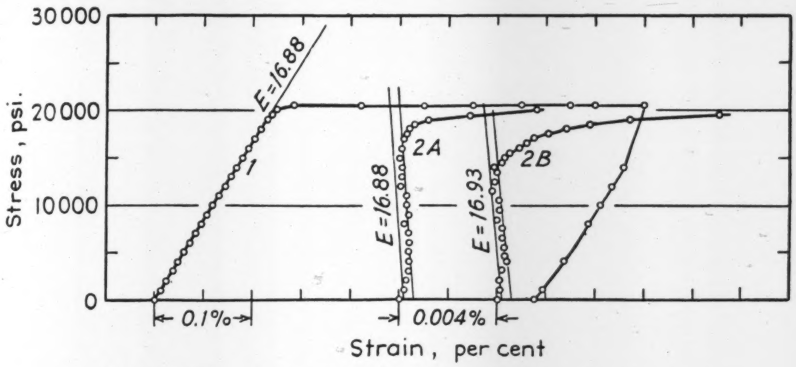

FIG. 12.—Stress-Strain Behavior of Annealed Phosphor Bronze C.

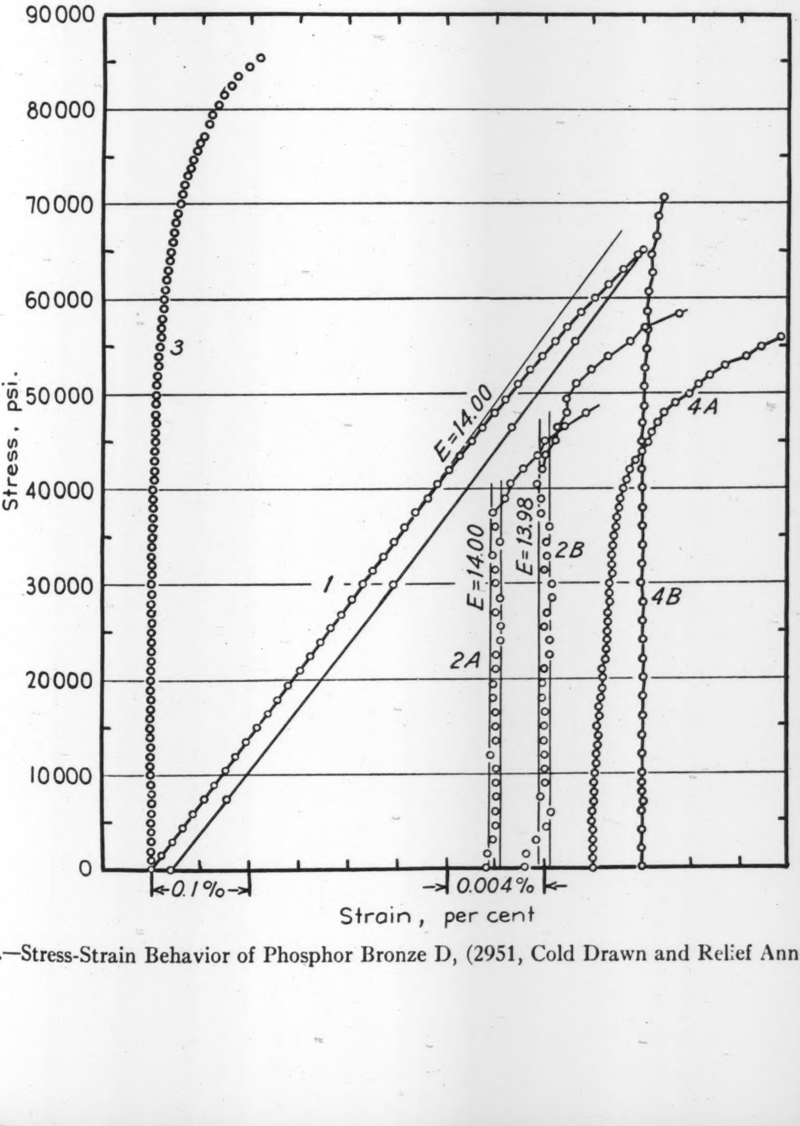

FIG. 13.—Stress-Strain Behavior of Phosphor Bronze D, (2951, Cold Drawn and Relief Annealed).

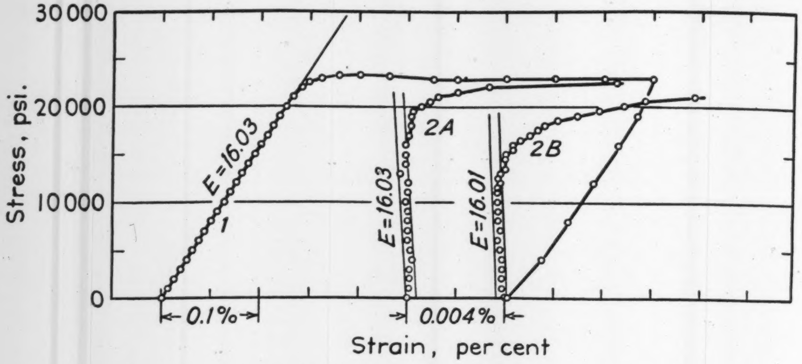

FIG. 14.—Stress-Strain Behavior of Annealed Phosphor Bronze D.

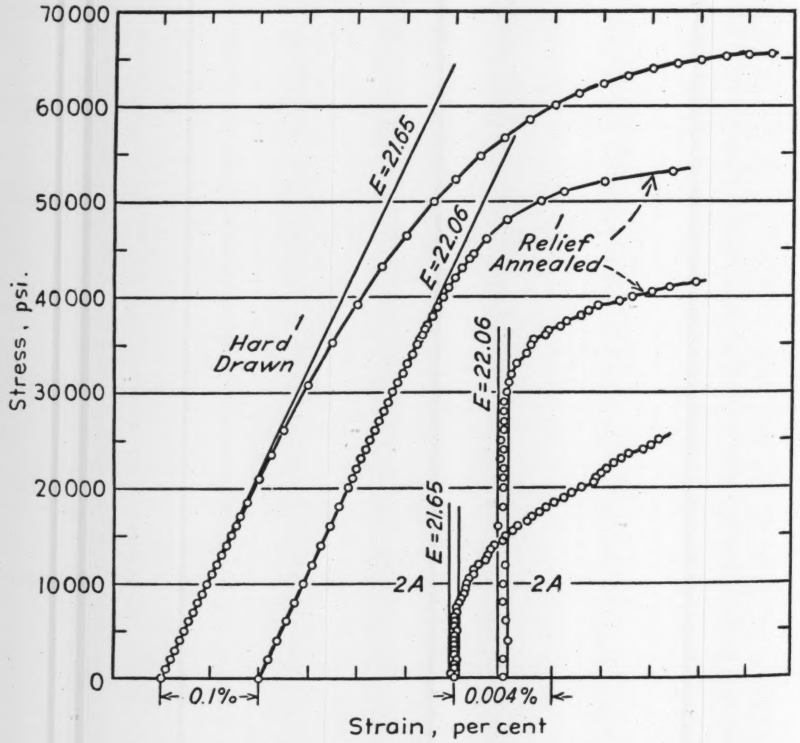

FIG. 15.—Stress-Strain Behavior of Cupronickel (3421) in Cold Drawn Condition and After Relief Annealing at 450 C.

detectable permanent set is greater than that for an equivalent offset, sometimes much greater as in the case of Everdur (Fig. 18). It is a curious fact, hard to explain, that phosphor bronze D and both varieties of copper (Figs. 1, 3, and 13) show permanent set at a stress lower than that which will produce measurable offset. Prestraining in tension invariably raises the stresses needed to produce a specified permanent set.

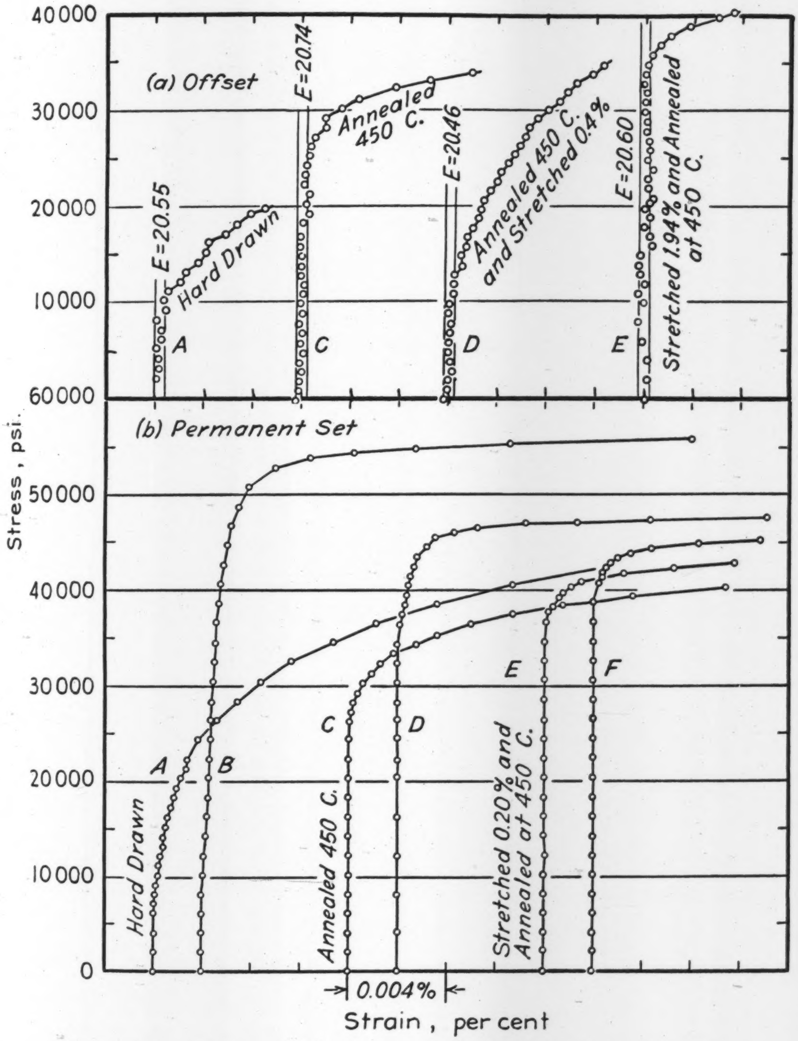

FIG. 16.—Curves Showing Ambrac (3420) Stressed In Various Conditions.

Prestraining tends slightly to increase the proportionality range of commercially drawn materials, though the high zinc alloys are exceptions. This effect is probably associated with the particular distribution of residual stresses in the cold-drawn rods and would vary greatly with manufacturing variables, particularly the final straightening operation.

Prestraining the annealed alloys always results in a marked decrease of the stress corresponding to 0.0002 per cent offset. Contrariwise, both varieties of copper have their proportionality range definitely increased by prestressing. Different alloys differ greatly in their behavior. Thus, while in annealed brass and Everdur the 0.0002 per cent offset stress is halved by prestraining, in the tin bronzes the decrease is only about ten per cent. The reason for this behavior is obscure. Internal friction measurements[4] show that related effects occur in the region of very small strains.

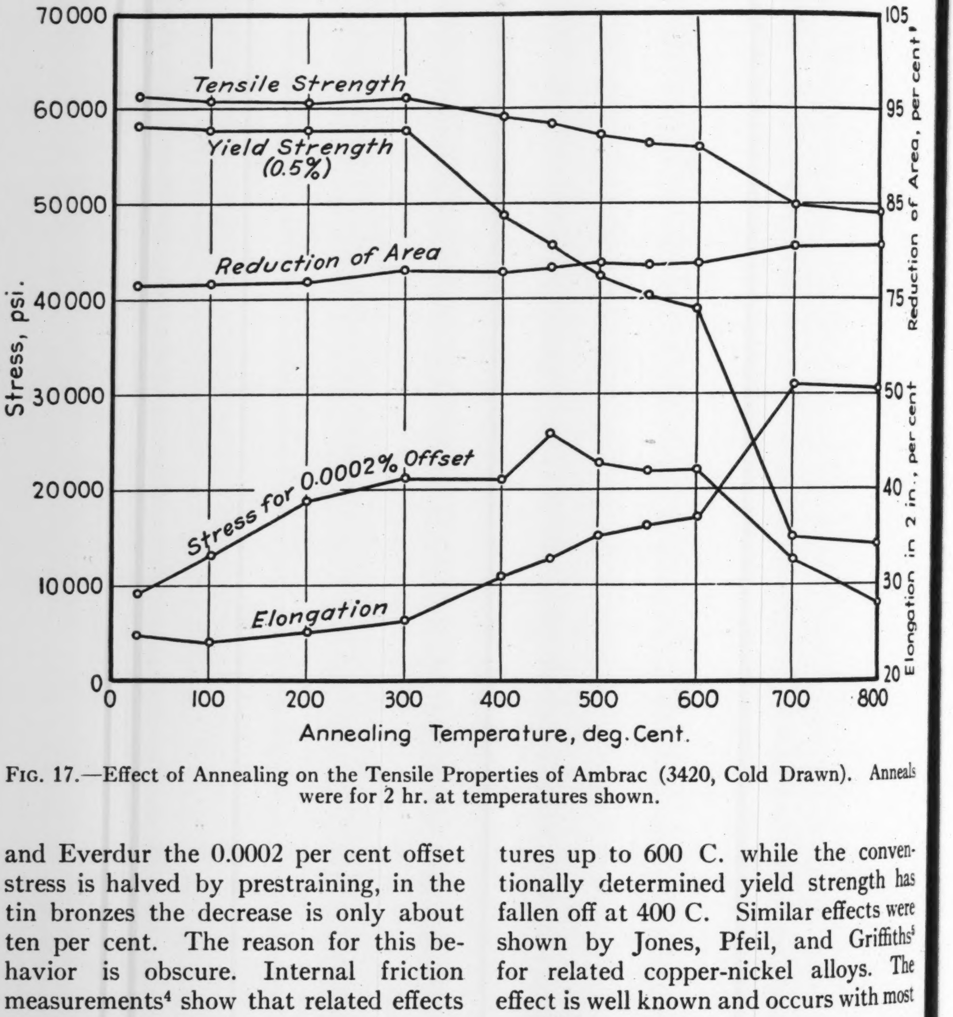

FIG. 17.—Effect of Annealing on the Tensile Properties of Ambrac (3420, Cold Drawn). Anneals were for 2 hr. at temperatures shown.

The effect of heat treatment on the properties of Ambrac (a copper-nickel-zinc alloy) is shown in Fig. 17. The stress corresponding to 0.0002 per cent offset is increased by heating at temperatures up to 600 C. while the conventionally determined yield strength has fallen off at 400 C. Similar effects were shown by Jones, Pfeil, and Griffiths[5] for related copper-nickel alloys. The effect is well known and occurs with most metallic materials.

Figure 16 shows permanent set and

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[4] W. Köster and K. Rosenthal, "Die Änderung von Elastizitätsmodul und Dämpfung bei der Verformung und Rekristallisation von Messing," *Zeitschrift für Metallkunde*, Vol. 30, pp. 345-348 (1938).

T. A. Read, "Internal Friction of Single Crystals of Copper and Zinc," *Technical Publication No. 1309*, Am. Inst. Mining and Metallurgical Engrs. (1941).

[5] D. G. Jones, L. B. Pfeil, and W. T. Griffiths, "Nickel-Copper Alloys of High Elastic Limit," *Journal*, Inst. Metals, Vol. 46, pp. 423-440 (1931).

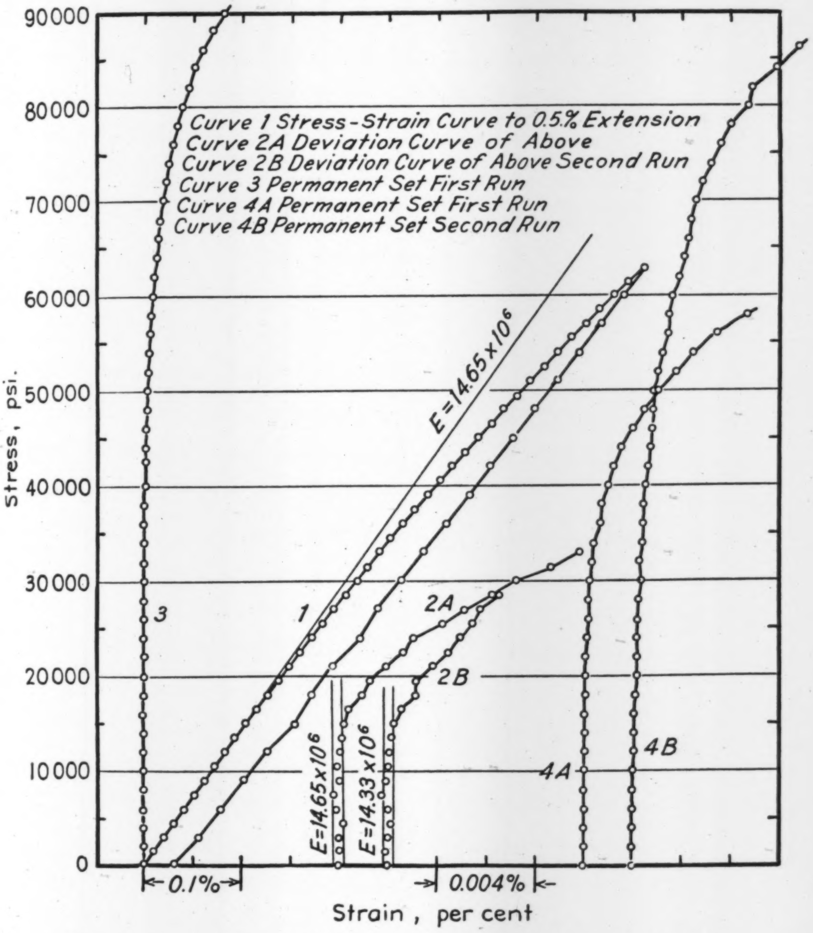

FIG. 18.—Stress-strain Behavior of Everdur 1010 (2901, Cold Drawn).

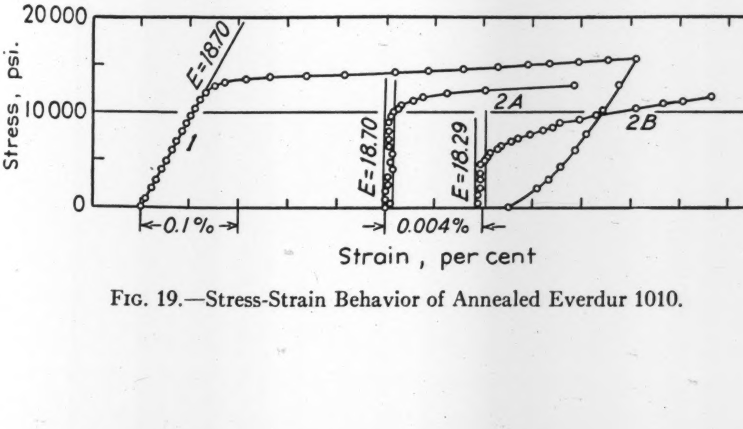

FIG. 19.—Stress-Strain Behavior of Annealed Everdur 1010.

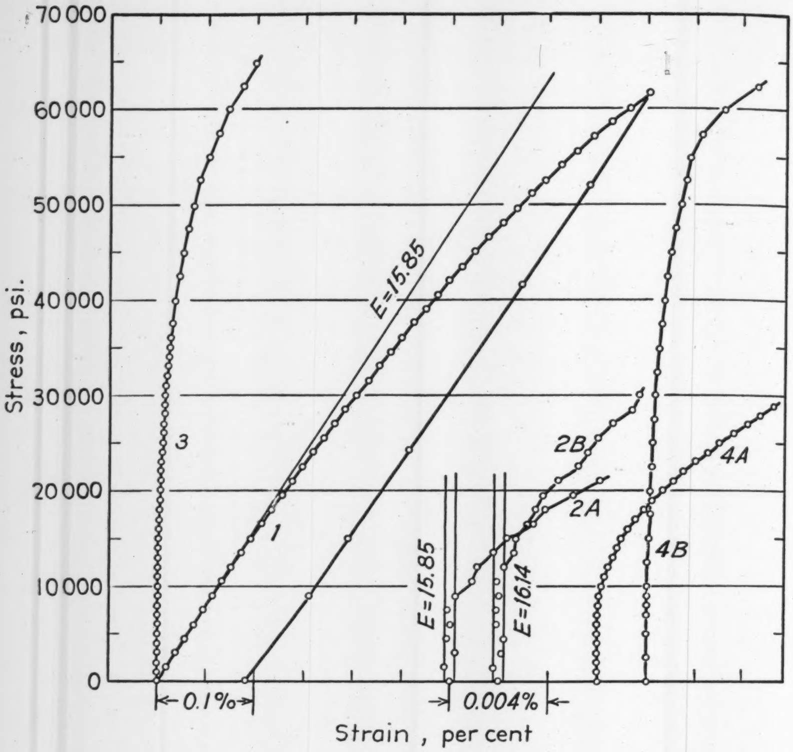

FIG. 20.—Stress-Strain Behavior of Everdur 1015 (2954, Extra Hard Drawn).

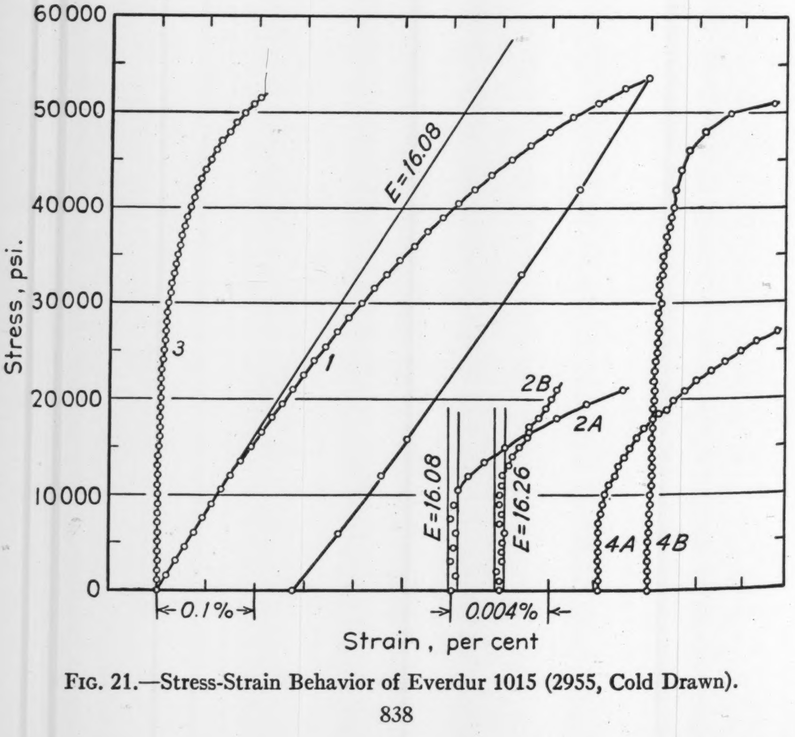

FIG. 21.—Stress-Strain Behavior of Everdur 1015 (2955, Cold Drawn).

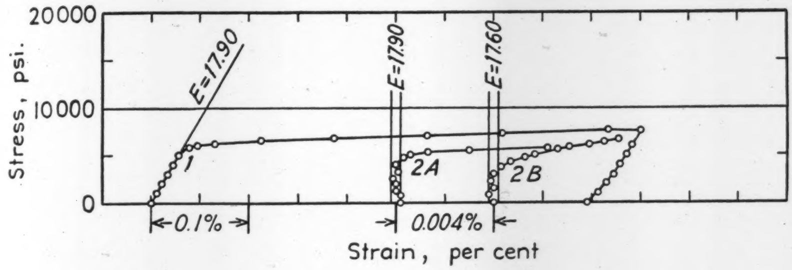

FIG. 22.—Stress-Strain Behavior of Annealed Everdur 1015 (2955).

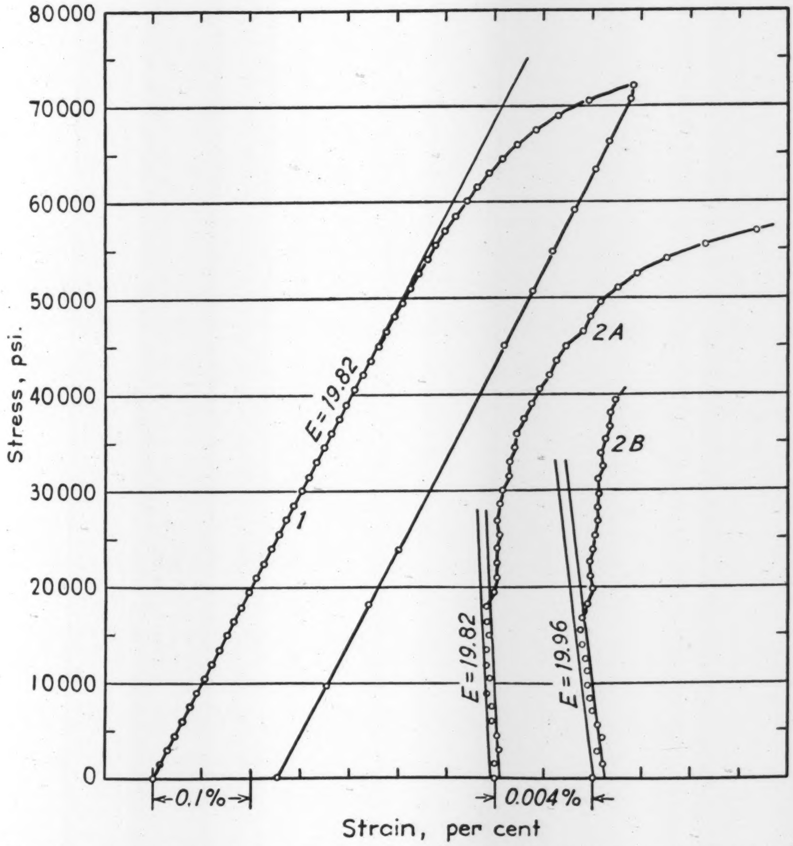

FIG. 23.—Stress-Strain Behavior of Chromium Copper (3960 HT, Quenched, Cold Drawn and Heat Treated).

deviation curves for this alloy stressed in various conditions, namely, (*A*) as drawn, (*B*) as *A* after straining to 0.10 per cent set, (*C*) after annealing at 450 C., (*D*) after annealing and applying a slight over-strain, (*E*) after straining in tension and then annealing, and (*F*) as *E* after stretching to 0.10 per cent set. A comparison between Fig. 16 (*a*) and (*b*) shows that permanent set and offset are quite different in the hard drawn and stretched conditions, but are approximately the same after relief annealing. It will be noticed that straining in tension followed by a relief anneal gives the highest range of elasticity in tension. This effect is probably a result of redistribution and partial retention of appropriately oriented microstresses. It suggests that instrument springs, Bourdon tubes, and other elastic measuring members, which are usually given a low-temperature anneal in the fabricated condition, would better be given a small over-strain by the application of stress in the direction of service stresses before the stabilizing heat treatment is applied. Moreover, it is obvious from Fig. 16 curve C that subsequent over-strain ruins the elastic properties of relief annealed material. Elastic measuring members should be so designed that testing overloads or service stresses never exceed the initial elastic limit.

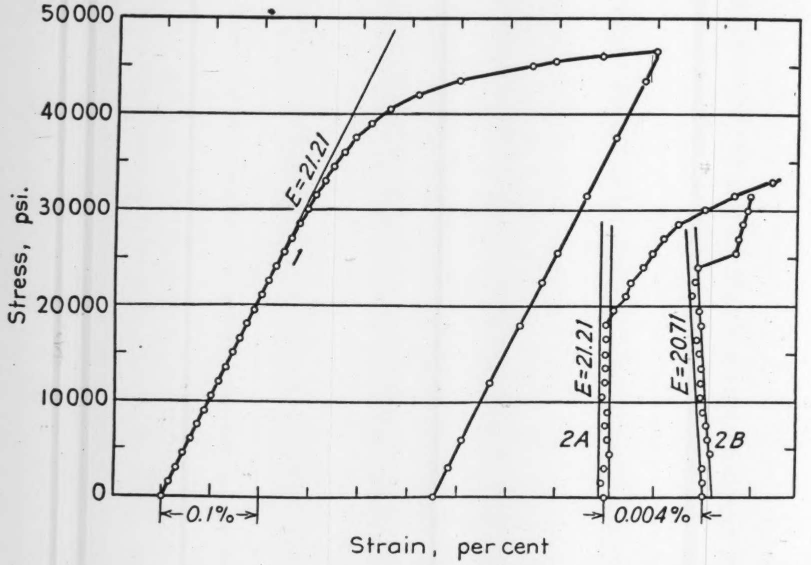

FIG. 24.—Stress-Strain Behavior of Chromium Copper (3960 AT, Quenched and Heat Treated).

BERYLLIUM COPPER

Beryllium copper is so outstanding in its characteristics as to merit special discussion. Figure 25 shows stress-strain curves in five different conditions and Fig. 26 the corresponding deviation curves for four of these. Figure 27 shows permanent set of the alloy after the various treatments. In the quenched or quenched-and-cold-drawn condition beryllium copper is slightly superior to the 10 per cent tin bronze. After precipitation heat treatment, its elastic properties are enormously improved.

This alloy illustrates well the importance of the corrections for true stress and true strain (that is, change of dimensions of the specimen under load) calculated by Anderson.[6] In curve *b*, Fig. 26, are shown the uncorrected experimental deviations from an initial modulus line; curve *a* shows the appropriate corrections for true strain based on Poisson's ratio (μ) = 0.35 and E = 18,400,000 psi.; curve *c* shows the data of curve *b* corrected for true strain (that is *b* minus *a*). The curvature of the uncorrected deviation curve is appreciable at stresses as low as 30,000 psi., but the point of departure from Hooke's law in terms of true stress and true strain is at about 100,000 psi. Obviously, the latter has some real significance whereas the "proportional limit" as normally obtained is nothing more than a mathematical function of E and μ and bears no relation whatever to the other properties of the alloy.

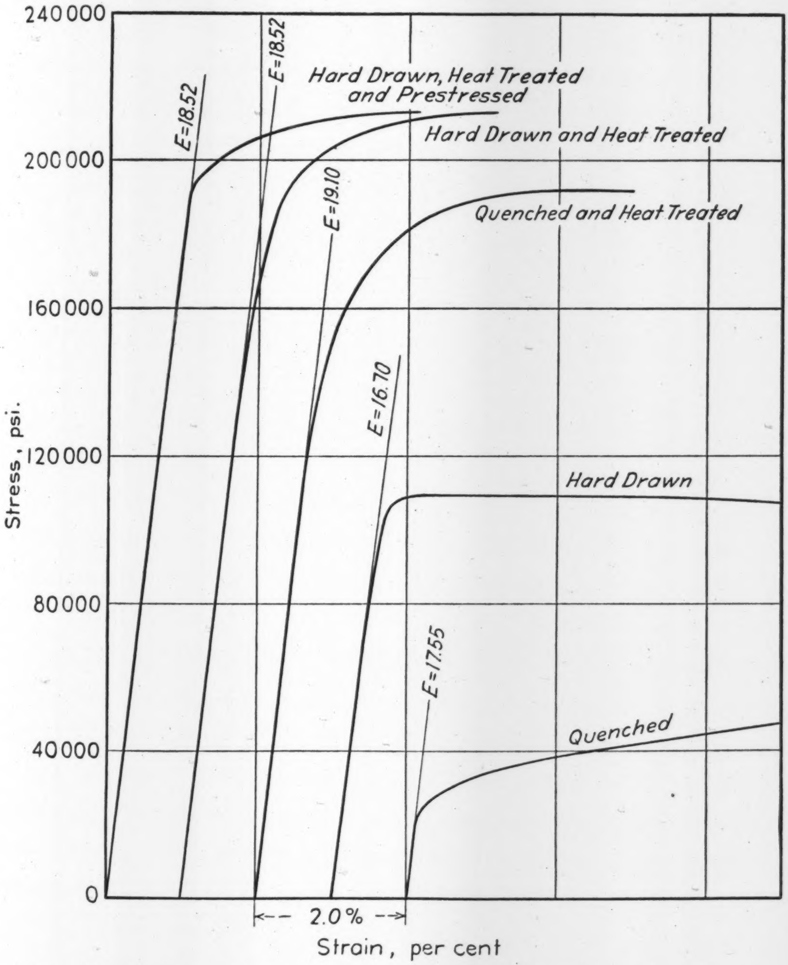

FIG. 25.—Stress-Strain Curves of Beryllium-Copper in Various Conditions.

Two series of stress-strain loops, made with a Ewing extensometer sensitive to 0.001 per cent, are shown in Figs. 28

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[6] Discussion by A. R. Anderson of paper by C. S. Smith, "Proportional Limit Tests on Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 877 (1940).

and 29. The curves show successive readings on specimens—quenched and heat treated in Fig. 29; quenched, cold drawn and heat treated in Fig. 28—taken to 0.2 per cent offset, then the stress released, reapplied to the same value, released, and again applied to produce a new 0.2 per cent offset and once more released. The fact that the return curve is nearly parallel to the ascending modulus and that reapplication of the same stress gave a narrow closed loop confirms the observation of Figs. 26 and 27, that offset and set are nearly equivalent, even in the realm of small strains. The alloy, moreover, seems to be immune from elastic damage by overstrain. Curves not reproduced here show that measurable departure from true proportionality occurs at 95,000 to 100,000 psi. with the cold-drawn and heat-treated material either initially or after prestraining. If this stress is not exceeded, the ascending and descending parts of the curve are superimposed while if this stress is exceeded, a closed loop of measurable width is formed, though there is no permanent

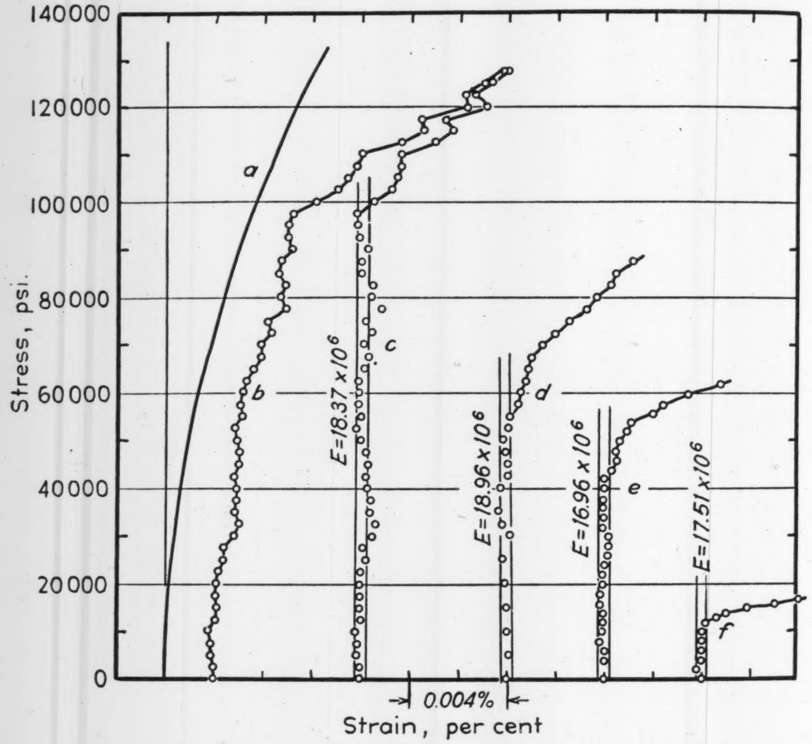

FIG. 26.—Curves Showing Deviation from Proportionality in Beryllium-Copper in Various Conditions.

a = correction for true stress and strain (E = 18.4 × 10^6 psi, μ = 0.35).
b = uncorrected deviation from E = 18.37 × 10^6 psi. for quenched, cold drawn and heat treated beryllium copper.
c = data of curve *b* corrected for true stress and true strain.
d = corrected deviation curve for quenched and heat-treated, beryllium copper.
e = corrected deviation curve for quenched and cold drawn beryllium copper, without heat treatment.
f = deviation curve for beryllium copper in quenched condition.

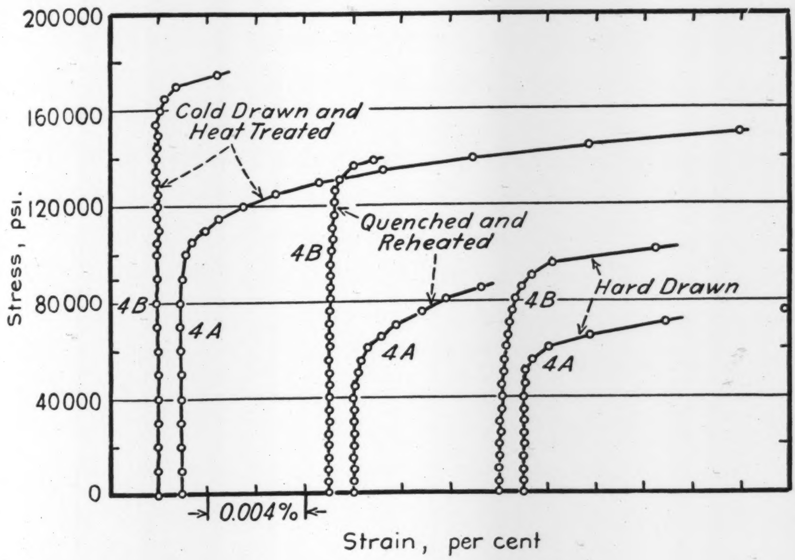

FIG. 27.—Permanent Set for Beryllium Copper After Various Treatments.

4A = tests on virgin materials.
4B = tests on material prestrained to 0.1 per cent permanent set.

TABLE III.—VALUES OF STRESS, STRAIN, SET AND OFFSET CORRESPONDING TO VARIOUS YIELD STRENGTH CRITERIA.

	0.5 per cent Total Strain			0.1 per cent Set	0.1 per cent Offset		0.2 per cent Offset	
	Stress at 0.5 per cent Strain, psi.	Offset at 0.5 per cent Strain, per cent	Resulting Permanent Set, per cent	Stress for 0.1 per cent Permanent Set, psi.	Stress, psi.	Total Strain, per cent	Stress, psi.	Total Strain, per cent
Electrolytic copper	46 000	0.229	0.212	44 300	43 700	0.359	45 700	0.473
O.F.H.C. copper	47 000	0.232	0.222	45 000	44 600	0.355	46 800	0.468
65-35 brass	56 500	0.160	0.109	57 500	51 700	0.412	58 700	0.552
Tobin bronze	32 300	0.273	0.224	29 600	29 300	0.306	31 400	0.421
Phosphor bronze A	60 400	0.141	0.121	61 800	57 800	0.443	63 000	0.575
Phosphor bronze C	60 900	0.094	0.077	65 200	61 700	0.510	69 400	0.662
Phosphor bronze D	65 600	0.030	0.019	84 500	79 900	0.674	87 400	0.828
Everdur 1010	62 900	0.074	0.012	92 500	73 800	0.600	90 800	0.815
Everdur 1015 XH	62 100	0.108	0.082	64 900	60 900	0.484	72 600	0.658
Everdur 1015 H	53 700	0.174	0.137	51 300	48 500	0.398	55 000	0.537
Beryllium copper HT	91 900	0.004	0.000	174 000	171 200	1.025	184 200	1.252
Beryllium copper AT	94 000	0.008	0.006	138 500	140 300	0.870	153 200	1.002
Chromium copper AT	46 500	0.280	0.277	....	43 500	0.306	45 600	0.426
Chromium copper HT	74 100	0.133	0.127	....	71 300	0.458	74 000	0.570

All alloys in hard condition unless otherwise noted.
For composition and other properties of alloys, see Tables I and II.

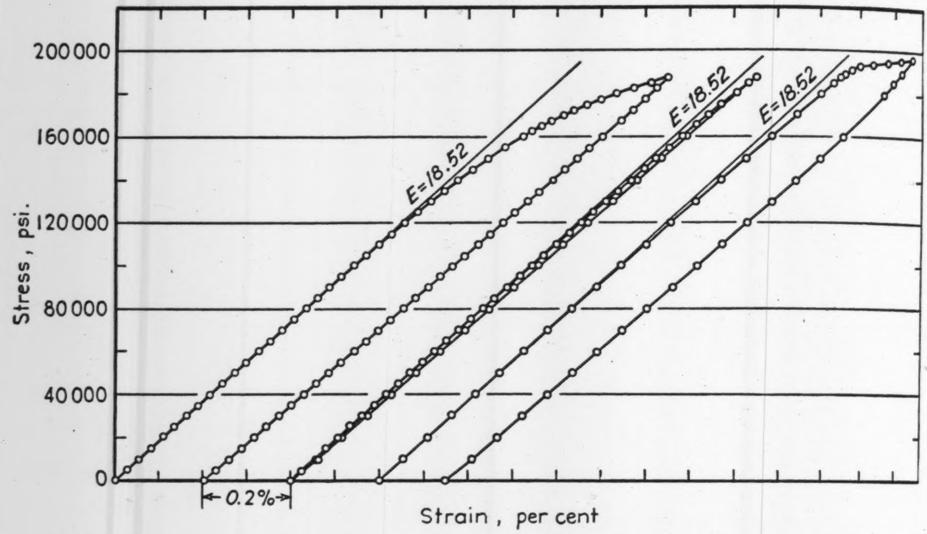

FIG. 28.—Stress-Strain Behavior of Beryllium Copper (Quenched, Cold Drawn and Heat Treated). Successive cycles of stress applied to same specimen.

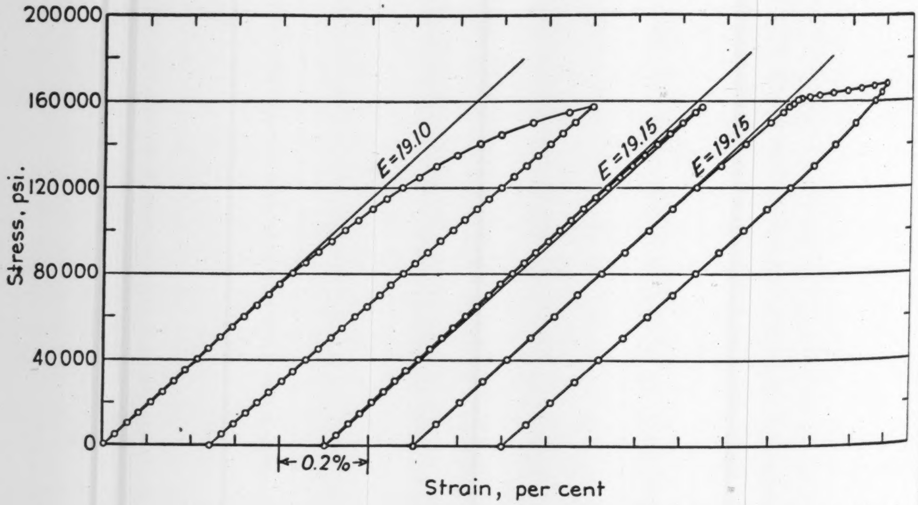

FIG. 29.—Stress-Strain Behavior of Beryllium Copper (Quenched and Heat Treated). Successive cycles of stress applied to same specimen.

set up to about 160,000 psi. in the once strained material.

YIELD STRENGTH MEASUREMENTS IN COPPER ALLOYS

It is customary in the copper industry to use a yield strength defined as the stress producing a total strain of 0.5 per cent. This is very satisfactory for acceptance testing (except possibly in the case of high-strength alloys) but it is unsuited for a comparison of the load-bearing behavior of materials of different moduli and yield strengths. Table III shows the offset and permanent set that results from stressing some copper alloys to the 0.5 per cent yield strength, and includes also a number of other possible criteria of plastic damage. The 0.5 per cent yield strength bears no direct relation to any index of plastic deformation, since there is an amount of elastic deformation that varies with the modulus and the applied stress. While for soft annealed materials the elastic contribution to strain is small, for hard phosphor bronze the elastic deformation is more than half the total, and in beryllium copper the entire specified 0.5 per cent extension can be produced elastically.

The value of the convenient single-strain method for specification testing is not questioned. The 0.5 per cent yield strength is, however, unsuited as a criterion on which to base possible service stresses for different materials, since it is not related to plastic damage. A more logical basis for establishing unit design stresses and for comparing the stress-resisting capacity of different materials would seem to be the stress producing some stated value of permanent set.

The engineer might be cautioned regarding the significance of the precise tests reported herewith. Slight departure from Hooke's law is without practical significance and without relation to possible service stresses. Small offsets are rarely accompanied by equivalent permanent sets. In another paper to be presented at the present meeting of the Society[7] the fatigue properties of a number of copper alloys are reported and it can be seen that these bear no consistent relation to offset or any other statically determined point.

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[7] A. R. Anderson and C. S. Smith, "Fatigue Tests on Some Copper Alloys," see p. 849.

DISCUSSION

MR. J. R. FREEMAN, JR.[1] (*presented in written form*).—The primary purpose of the work presented in this paper and the previous one published last year[2] is to make available to engineers reliable data on the true elastic properties of copper alloys suitable for design purposes. I think the authors deserve great credit for the development of the exceedingly precise test methods used and the carrying through of the very painstaking and time-consuming test program necessary to obtain the detailed data presented. It is hoped that the test program may be continued to obtain data on the same alloys at different tempers and other copper-base alloys of engineering interest.

I should like to direct particular attention to the discussion of yield point measurements in the latter part of the paper. The yield strength value, defined as the stress producing a total strain of 0.5 per cent under load, has become the standard method adopted by the copper alloy industry for specification purposes. It is used as the basis for defining the temper or degree of cold work in an alloy in practically all A.S.T.M., commercial, and Government specifications. It has proved to be entirely satisfactory for the purpose of acceptance testing, with the exception of a few very special cases where the alloy has exceptionally high elastic strength.

The data presented in the paper confirm what is well known, namely, that the 0.5 per cent yield strength value bears no direct relation to permissible design stress. The recommendation has therefore been made by some engineers and specification writers that a definite permanent set or offset value should be used in commercial acceptance testing, such as the 0.2 per cent offset value used for aluminum alloys. I would call attention to the comparison of stress values given in Table III for yield strength at 0.5 per cent strain, and stress at 0.2 per cent offset. It will be noted that for all the alloys tested the latter stress is approximately the same as the former, or appreciably greater, the increase in stress value being more marked for the stronger alloys. Further, even the 0.2 per cent offset value does not give an exact basis for design stresses (which are properly based on values for permanent set) as is evident on comparing the stress values for a given offset in Table III with values for permanent set given for the same alloys in Table II.

The design engineer, therefore, must use empirical values for factors of safety if he uses either the 0.5 per cent strain, the 0.2 per cent offset or, in fact, any offset value. His most reliable criterion is permanent set. The determination of the stress required to produce a stated permanent set in a test specimen is a lengthy and relatively costly procedure that cannot be used in the routine testing laboratory for acceptance testing. The determination of a definite offset value requires the plotting and drawing of a stress-strain curve or use of special automatic curve-drawing equipment not available in many laboratories. It is definitely more time consuming than the method of determining 0.5 per cent strain under load. This latter method is quick, inexpensive and has proved reliable for many years in general acceptance testing. Once the stress value for a given permanent set has been established for a material with known temper or properties as defined by the yield strength for 0.5 per cent strain under load, other lots of the same material produced by the same standard practices will show the same stress value for a given permanent set. The continued use of the 0.5 per cent strain under load value for acceptance testing is therefore fully justified.

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[1] Technical Manager, American Brass Co., Waterbury, Conn.

[2] Cyril Stanley Smith, "Proportional Limit Tests on Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 864 (1940).

MR. CYRIL STANLEY SMITH[3] (*author's closure, by letter*).—Mr. Freeman's discussion on the significance of yield strength in relation to acceptance testing and design is most welcome. Too many engineers confuse the divergent requirements of research testing for the purpose of establishing design stresses with those of routine testing for the purpose of finding whether a new batch of material is as good as the previous lot. It seems to me that once the fundamental difference between these two types of test is realized the problem of obtaining satisfactory acceptance tests is considerably simplified. Tests such as those described in the present paper are necessary for an understanding of the behavior of a material under stress, but they are not necessary for acceptance tests. No one would think of redesigning a piece of equipment to utilize the exact strength of each particular bar of metal, and an acceptance test does not need to provide such a figure. It seems to me that an acceptance yield strength should be defined as the stress that produces a plastic strain that is easily measurable but not too high, and, while this strain must obviously be the same for successive batches of a particular material or temper, it is immaterial whether it is the same for all materials or not. For this reason I agree fully with Mr. Freeman that the present yield strength corresponding to 0.5 per cent total elongation is adequate for specification testing of most copper alloys. I would, however, limit its use to the determination of yield strengths that do not exceed 60,000 psi., for at that point (with material having a modulus of 15 million psi.) the offset becomes 0.1 per cent, and under present conditions of commercial testing this is about as small a plastic deformation as can be measured with the desired accuracy. For stresses above this point, I would suggest that a strain of 0.75 per cent be employed, up to the stress where this, too, ceases to correspond to more than 0.1 per cent offset (97,500 psi.), when the strain would again be changed to 1.0 per cent. In this manner the plastic strain involved would always be at least 0.1 per cent and would never be over 0.6 per cent for any material as long as the modulus falls within the range 15 to 20 million psi., which includes the majority of the copper alloys. This system has the great advantage that it involves no change for alloys with yield strengths below 60,000 psi., and above this it continues to employ the advantages of the present system. The appropriate strain is the same for all copper alloys of a given yield strength and is at once apparent from the stress figure. Being a round number, it is easily read on the extensometer. The present mass of experience data continues to be of value, for the majority of alloys will still be tested at 0.5 per cent extension.

It has been proposed as a modification of the 0.2 per cent offset yield strength to find the stress that produces a total strain equivalent to the minimum 0.2 per cent offset point determined by prior trial. This has a disadvantage that each material and each temper requires a different strain and this strain will be an odd number, hard to remember and hard to read on the extensometer dial. With inevitable changes of modulus of elasticity and with the nonaxiality of loading that exists in routine testing, I question whether such refinement achieves its purpose and think that equally valuable results could be obtained by the use of the simpler round-number strains advanced above.

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[3] Research Metallurgist, American Brass Co., Waterbury, Conn.

Finally, I wish to repeat that it must be realized that the yield strength determined for specification purposes is not intended to define the properties of the material but merely to ascertain if the material has the same stress-resisting characteristics as a previous lot. For a comparison between different materials and for establishing design stresses, the engineer needs far more complete data, and it is suggested that actual stress-strain curves together with tabulated values of stress for certain standard sets, offsets, and strains could with advantage be included as an informative appendix to all metal specifications.

FATIGUE TESTS ON SOME COPPER ALLOYS

By A. R. Anderson[1] and Cyril Stanley Smith[2]

Synopsis

The paper presents the results of short-specimen rotating-beam endurance tests on commercially fabricated rods of tough pitch and oxygen-free copper and fifteen copper alloys, including precipitation-hardening types. Endurance strengths, at 300 million cycles, are tabulated, together with pertinent data on composition, fabrication and tensile properties, while the experimental data are presented in the form of *S-N* curves. The results are briefly discussed and the fatigue machines, preparation of specimens, and testing technique are described.

The large amount of published data on the endurance properties of steels is in marked contrast with the paucity of information on copper alloys. In 1932 Gillett (**1**)[3] adequately summarized the information then available on the endurance properties of copper alloys. Since that time important papers have been published by Gough and Sopwith (**2, 3, 4, 5**)(mainly concerned with atmospheric and other corrosion factors in fatigue), by Greenall and Gohn (**6**), and by Price and Bailey (**7**) (describing the endurance properties of commercial copper alloys in the form of cold-rolled strip). Kommers (**8**) studied the effect of straining and low temperature annealing on brass; Boone and Wishart (**9**) studied brass at sub-atmospheric temperatures, and Krouse (**10**) investigated the speed effect with brass.

The present determinations were made on carefully polished samples machined from commercially hard-drawn rods, and the values obtained are usually higher than those of investigators who utilized strip as commercially furnished in the rolled condition. The figures approximate ideal endurance strengths (at 300 million cycles in air) from which the effect of various factors such as notches, corrosion and the like may later be evaluated. Pertinent information regarding composition, fabrication, structure and other properties is recorded.

Testing Procedure

All tests were performed in rotating-beam machines of the type shown in Fig. 1. Fourteen such machines were used interchangeably. They differ from the short-specimen Farmer machine primarily in the method of applying load and the driving mechanism. The load is suspended from the bottom of the housings of self-aligning ball bearings, a design which leads to simplicity and cheapness of construction. In order to minimize vibration, the driving torque is applied through a spring and a bayonet

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[1] Research Assistant, Copper Alloys Research Laboratory, American Brass Co., Waterbury, Conn.

[2] Research Metallurgist, American Brass Co., Waterbury, Conn.

[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 857.

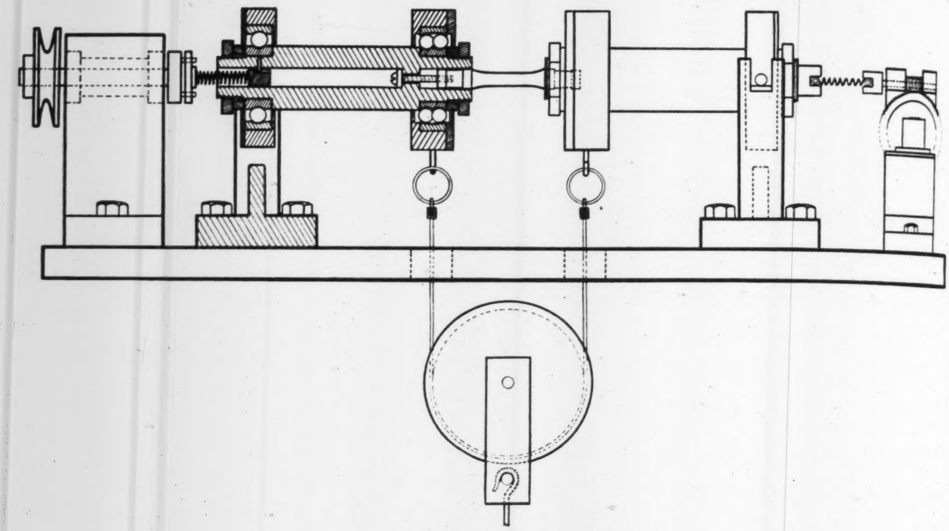

FIG. 1.—Fatigue Testing Machine.

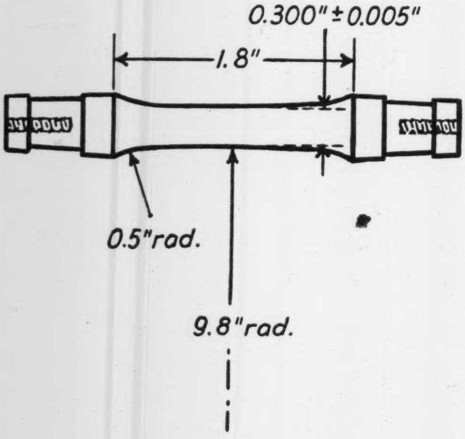

FIG. 2.—Fatigue Specimen.

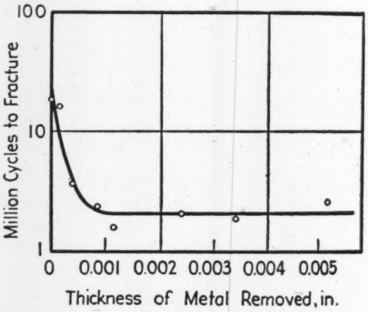

FIG. 3.—Effect of Amount of Metal Removed, by Abrasive Polishing, on Life of Annealed Everdur Specimens (3198-A) at 21,500 psi. Stress.

catch to a pin located inside the spindle at the center of the left-hand bearing.

Most materials were tested at a speed of 3500 rpm., though some were tested at 1600 rpm. A bronze tested at both speeds showed a negligibly small speed effect, as can be seen in Fig. 4 (*d*). Stresses were computed in the conventional manner, assuming elastic behavior. The uncertainty in the value of computed stress due to errors in measurements is believed to be less than 1 per cent.

Specimens of the R. R. Moore type, with the dimensions shown in Fig. 2, were machined from 0.5-in. or larger diameter rods. Other specimens from 0.398-in. diameter rods were of similar contour in the test portion but of minimum diameter, 0.250 ± 0.005 in. The contours of the gage lengths were produced by the use of suitably shaped guides for the lathe carriage. A pointed tool finishing with 2 or 3 cuts about 0.002 in. deep was used in an effort to

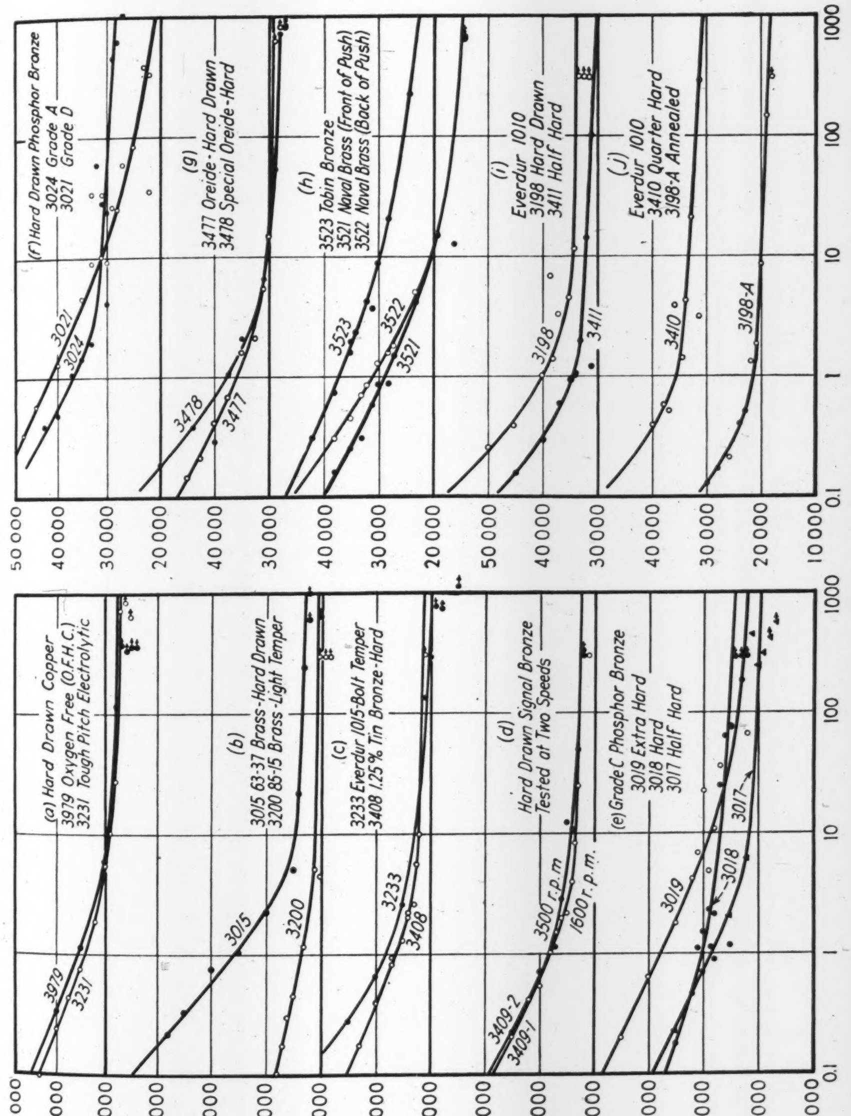

FIG. 4.—*S-N* Curves of Alloys Tested, Showing Experimental Points. Arrows indicate unbroken specimens.

TABLE I.

Alloy and Curve Number	Material	Commercial Temper Designation	Final Reduction of Area, per cent; or Anneal, deg. Cent.	Estimated Ready-to-Finish Grain Size, mm.	Composition, per cent							Endurance Strength at 300 million cycles, psi.	Modulus of Elasticity, psi. (× 10⁻⁶)	Stress, psi.			Yield Strength (0.5% Extension), psi.	Tensile Strength, psi.	Elongation, per cent		Reduction of Area, per cent	Rockwell Hardness	Notes	Alloy and Curve Number
					Copper	Tin	Zinc	Phosphorus	Iron	Lead	Other Elements			0.0002% offset	0.002% offset	0.2% offset			2-in. gage length	Gage length 4 times diameter				
3231.......	Tough pitch copper (N.E.C.)	Hard	36.0	0.040	99.948	....	....	....	....	....	O_2 0.031 S 0.0029	17 000	17.77	4 500	8 400	46 500	47 600	48 800	9.8	14.0	61.7	B46.6	(1) (6)	3231
3979.......	Oxygen-free copper (O.F.H.C.)	Hard	29.2	0.125	99.997	....	....	....	....	....	O_2 0.000 S 0.0016	17 000	17.80	6 000	9 000	49 000	49 400	51 000	14.0	21.6	88.1	B37.2	(1) (6)	3979
3200.......	85-15 brass	Light	4.0	0.025	84.53	....	15.44	....	0.02	0.01	....	20 000	17.15	7 000	10 400	28 200	29 300	44 700	40.0	49.1	80.5	B50.5	(1)	3200
3015.......	63-37 brass	Hard	30.1	0.060	63.00	....	36.96	....	0.01	0.03	....	22 700	15.72	17 600	26 000	55 500	53 900	72 200	17.8	24.4	61.6	B81.6	(1)	3015
3408.......	1.25% tin bronze	Hard	36.6	0.015	98.68	1.25	0.00	0.05	....	....	....	31 300	19.00	24 000	32 100	54 800	54 700	59 200	14.2	23.3	74.5	B69.5	(2) (4)	3408
3409-1.....	Signal bronze	Hard	36.6	0.015	98.37	1.59	0.00	0.03	....	....	....	32 700	18.98	28 300	35 300	57 300	57 200	62 400	14.8	23.0	73.2	B71.0	(2) (4)	3409-1
3409-2.....	Signal bronze	Hard	36.6	0.015	98.37	1.59	0.00	0.03	....	....	....	32 700	18.98	28 300	35 300	57 300	57 200	62 400	14.8	23.0	73.2	B71.0	(4) (9)	3409-2
	Phosphor bronze:																							
3024.......	Grade A	Hard	30.1	0.090	95.27	4.32	0.00	0.38	0.025	0.003	....	29 000	16.38	35 600	39 800	61 600	60 000	69 800	22.7	32.5	78.7	B91.5	(1) (4)	3024
3019.......	Grade C	Extra hard	50.1	0.100	91.78	8.10	0.00	0.08	0.043	0.002	....	30 000	13.80	34 300	43 600	93 100	65 300	110 300	10.0	14.7	69.2	B98.0	(1) (4)	3019
3018.......	Grade C	Hard	30.1	0.100	91.78	8.10	0.00	0.08	0.043	0.002	....	34 700	15.00	36 000	39 700	64 500	60 200	81 000	26.7	35.2	77.3	B96.5	(1) (4)	3018
3017.......	Grade C	Half hard	15.2	0.100	91.78	8.10	0.00	0.08	0.043	0.002	....	30 000	16.43	30 000	33 000	49 100	49 100	66 000	47.8	56.5	81.1	B80.2	(1) (4)	3017
3021.......	Grade D	Hard	30.1	0.090	89.86	9.76	0.20	0.12	0.06	0.00	....	23 000	14.07	32 800	35 300	70 400	60 000	92 100	23.2	30.4	63.6	B91.4	(1) (4)	3021
3477.......	Oreide	Hard	30.1	0.035	88.47	1.02	10.49	....	0.02	0.00	....	29 300	17.04	18 800	32 100	61 800	59 900	65 200	12.2	19.5	76.6	B79.9	(1) (4)	3477
3478.......	Special oreide	Hard	30.1	0.040	87.02	1.10	11.48	0.37	0.02	0.01	....	28 300	15.75	22 500	36 100	69 200	62 900	78 800	12.8	19.7	63.7	B88.9	(1) (4)	3478
3234.......	Tobin bronze	Hard	9.4	See text	60.09	0.67	39.16	....	0.02	0.06	....	28 000	14.10	24 700	27 300	38 300	39 300	66 300	34.0	39.2	54.4	B79.5	(1)	3234
3523.......	Tobin bronze	Hard	11.5	See text	59.97	0.89	39.08	....	0.03	0.03	....	23 700	14.07	13 500	19 700	50 500	49 000	69 600	25.3	29.6	50.8	B76.9	(3)	3523
3521.......	Naval brass	Hard	11.5	See text	60.00	0.82	39.13	....	0.01	0.04	....	15 000	13.44	12 400	18 000	53 700	49 900	69 900	23.8	26.0	46.5	B75.6	(3)	3521
3522.......	Naval brass	Hard	11.5	See text	60.05	0.87	39.02	....	0.02	0.04	....	15 000	14.44	12 600	18 600	55 000	53 500	72 300	23.3	29.6	47.4	B81.7	(3)	3522
3198.......	Everdur 1010	Hard	39.0	0.125	95.49	....	....	....	0.15	....	Si 3.20 Mn 1.07	33 600	14.63	18 400	27 400	80 000	64 200	97 100	12.8	18.4	61.7	B100.7	(1)	3198
3411.......	Everdur 1010	Half hard	20.0	0.065	96.13	....	0.00	....	0.16	....	Si 2.91 Mn 0.95	30 500	....	....	....	....	49 100	74 900	36.7	....	....	B88.9	(2)	3411

3410.......	Everdur 1010	Quarter hard	8.0	0.035	96.13	....	0.00	....	0.16	....	Si 2.91 Mn 0.95	31 500	....	....	....	....	45 200	66 600	48.7	....	....	B83.9	(2)	**3410**
3198-A....	Everdur 1010	Soft	0.5 hr. 700 C.	0.085	95.49	....	....	....	0.15	....	Si 3.20 Mn 1.07	18 800	16.94	10 100	11 500	14 500	16 000	59 600	67.8	75.2	78.0	B43.6	(1) (5)	**3198-A**
3233.......	Everdur 1015	Bolt	71.6	....	98.17	....	0.00	....	0.06	....	Si 1.41 Mn 0.21	30 400	16.98	11 100	19 300	80 500	68 500	88 000	10.0	14.9	74.0	B86.0	(1)	**3233**
2991-H....	Beryllium copper	Heat treated	1.5 hr. 300 C.	....	97.54	....	....	....	0.07	....	Be 2.12 Ni 0.31	61 500	....	....	....	....	....	....	....	....	....	G99.1	(1)	**2991-H**
3419-H1...	Beryllium copper	Heat treated	1.5 hr. 300 C.	0.025	97.42	....	....	....	0.09	....	Be 2.12 Ni 0.40	55 000	18.8	71 000	83 500	....	93 500	186 900	5.0	6.7	18.9	G98.2	(1) (7) (4)	**3419-H1**
3419-H2...	Beryllium copper	Heat treated	1.5 hr. 300 C.	0.025	97.42	....	....	....	0.09	....	Be 2.12 Ni 0.40	47 200	....	....	....	....	....	....	....	....	....	G102.2	(1) (7)	**3419-H2**
3326.......	Chromium copper	Hard	91.8	....	98.80	....	....	....	0.07	....	Si 0.09 Cr 0.88	25 800	19.80	3 800	8 700	66 900	66 100	72 800	7.5	11.5	53.5	B72.9	(1)	**3326**
3326-H1...	Chromium copper	Heat treated	3 hr. 450 C.	....	98.80	....	....	....	0.07	....	Si 0.09 Cr 0.88	28 000	....	....	....	....	70 200	76 800	15.0	20.0	54.2	B82.4	(1) (8)	**3326-H1**
3326-H2...	Chromium copper	Heat treated	3 hr. 435 C.	....	98.80	....	....	....	0.07	....	Si 0.09 Cr 0.88	28 000	19.68	30 900	43 800	72 000	70 000	77 400	13.3	16.8	53.5	B82.0	(1) (8) (4)	**3326-H2**

NOTES:

(1) Tension specimens 0.313 in. in diameter, fatigue specimens 0.300 in. in minimum diameter; machined from 0.500-in. diameter rod. Fatigue tests run at 3500 rpm.

(2) Tension specimens 0.250 in. in diameter, fatigue specimens 0.250 in. in minimum diameter; machined from 0.398-in. diameter rod. Fatigue tests run at 1600 rpm.

(3) Tension specimens 0.313 in. in diameter, fatigue specimens 0.300 in. in minimum diameter; machined from 1.000-in. diameter rod; specimen axes ¼ in. from surface of rod. Fatigue tests run at 3500 rpm.

(4) Modulus of elasticity and 0.0002 per cent and 0.002 per cent off-set stresses obtained from deviation curves corrected for true stress and true strain.

(5) Grain size after final anneal.

(6) Copper figure includes silver.

(7) 21.0 per cent final reduction of area prior to precipitation heat treatment.

(8) 91.8 per cent final reduction of area prior to precipitation heat treatment.

(9) Same as (2), except fatigue tests run at 3500 rpm.

minimize work hardening of the specimen surface. In spite of this, appreciable work hardening or introduction of compressive stresses at the surface evidently took place, for a number of instances were observed where a machined specimen with tool marks as it left the lathe had a longer life, at a given stress, than did a carefully polished specimen of the same material. Figure 3 illustrates this for soft Everdur. About 0.001 in. of metal had to be removed by abrasive polishing before the effect of machining became insignificant. The magnitude of the effect in terms of the endurance strength was not determined.

Various polishing techniques were employed during the investigation. In all cases, however, the specimens were finished with a longitudinal polish using a suspension of Alundum 600 in kerosine or water. Specimens were gaged before completion of the final polish. The present preferred polishing practice is to remove lathe marks and 0.001 to 0.002 in. of the underlying material with kerosine-soaked 150 abrasive cloth ("Lightning Metalite") backed with a taut band of neoprene. A simple machine handling four specimens simultaneously reciprocates the polishing pads at 200 complete strokes per minute, while rotating the specimen 2 rpm. Longitudinal scratches from the coarse cloth are then completely removed by a circumferential polish using 320 cloth with kerosine, backed with sponge rubber and applied with light pressure. A final longitudinal polish in the machine using 600 Alundum on a broadcloth pad backed with leather and soaked with kerosine removes all traces of circum-

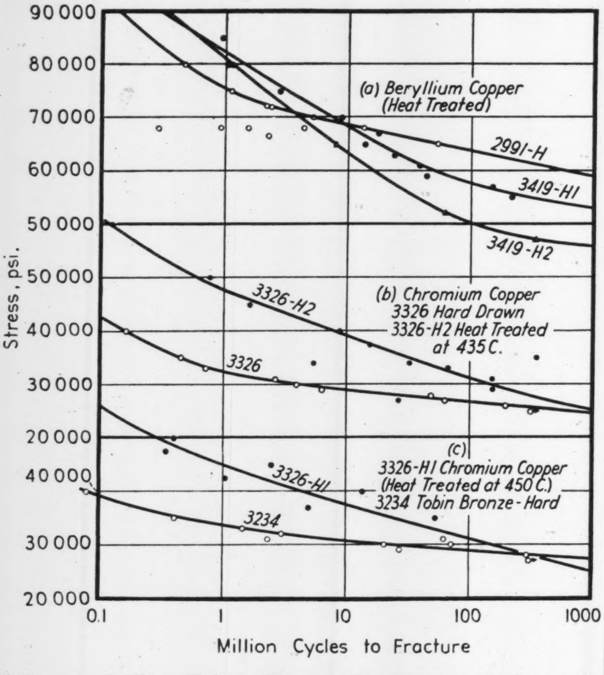

FIG. 5.—*S-N* Curves of Alloys Tested, Showing Experimental Points. Arrows indicate unbroken specimens.

ferential scratches. The finished specimen is then washed in kerosine and petroleum ether and coated with a few drops of white medicinal mineral oil.

Materials Tested

Various tempers of commercially produced copper and copper alloy rods were tested. The compositions are given in Table I, together with pertinent details of grain sizes, amounts of final cold work, final anneals or heat treatments, and a summary of mechanical properties. The tension tests were made using the refined technique previously described (11). The stress producing 0.0002 per cent offset represents the first consistently detectable departure from proportionality; the 0.002 per cent offset stress is roughly equivalent to the proportional limit that would be obtained in ordinary high quality commercial testing. True stress-true strain corrections (12) were applied, when appreciable, in obtaining these values.

Results of the endurance tests are presented in the form of *S-N* curves, plotted to a semilogarithmic scale, in Figs. 4 and 5. The curve drawn in each case is that which seems best to fit the experimental data, rather than the lowest possible curve. The endurance strengths at 300 million cycles listed in Table I represent values of computed maximum fiber stresses at the intersections of these *S-N* curves with the 300 million cycles ordinates.

Discussion

Most of the work-hardened solid-solution alloys tested need little comment. The results seem to be well established for the particular lots of material tested. The well-known effect of cold work in increasing endurance strength is shown for a phosphor bronze, Fig. 4 (*e*) and a silicon bronze, Fig. 4 (*i*) and (*j*). Comparatively little initial working raises the endurance strength rapidly, while further working produces less effect and may even reduce it, perhaps as a result of increased notch sensitivity. It is significant that "scatter" frequently increases with the amount of cold work.

Alloys whose structures vary with treatment show wide differences in endurance behavior. Naval brass is of alpha plus beta structure and the amount and distribution of the two phases, greatly affected by treatment, itself affects the endurance properties. Figure 4 (*h*) compares the behavior of specimens machined from two 1-in. rods, prepared by extrusion and by hot rolling, respectively, before the final cold drawing to size. The extruded material has a large grain size and varies in structure from front to back of the push. The hot-rolled material (commercial designation "Tobin Bronze") is of finer grain size, contains less beta and is generally more uniform. The difference in endurance strength is marked. Figure 5 (*c*) shows the behavior of material of similar composition made by hot rolling and cold drawing to a smaller size.

For the precipitation hardening alloys, the effect of precipitation heat treatment is not nearly so great on endurance strength as on tensile strength, and considerable scatter of points may be obtained. This is well illustrated by the curves for heat-treated chromium copper, Fig. 5 (*b*) and (*c*). Although heat treatment does not appreciably raise the endurance strength at 300 million cycles, it does greatly increase the life at higher stresses. The results are extremely sensitive to slight variations in heat treatment, which possibly accounts for much of the scatter. This can be seen from Fig. 6 wherein is plotted the life of a specimen at 40,000 psi. *versus* the temperature of its heat treatment for 3 hr. Maximum life is obtained after treatment at 435 C. A life only half as great results if the temperature varies 15 deg. in either direction, while the effect on Rockwell hardness is negligible. It does not necessarily follow, however, that this temperature results in maximum endurance strength at 300 million cycles.

Figure 5 (*a*) shows *S-N* data for beryllium-copper and again demonstrates the importance of heat treatment variations. Curve 3419-H1 is well defined by 11 points. Curve 3419-H2 is also well defined, though with fewer points, and is quite distinct from the former. Both curves were obtained on material that was identical, save for the final heat treatment, and this, performed in the laboratory with care, was intended to be the same. Curve 2991-H is on a different lot of material but supposedly the same. The considerable scatter of certain points seems directly attributable to observed variations in grain size of the metal prior to cold working, the variations being appreciable but within the amount to be expected from commercial fabrication. Taking any of the three curves as drawn, the endurance strength is considerably higher than earlier figures reported in the literature. It is not to be expected that commercial material will consistently reach even the minimum of these values—47,000 psi. Also, it should be pointed out that unpolished specimens of this material would be expected to show more scatter and fail at lower stresses than these carefully polished bars.

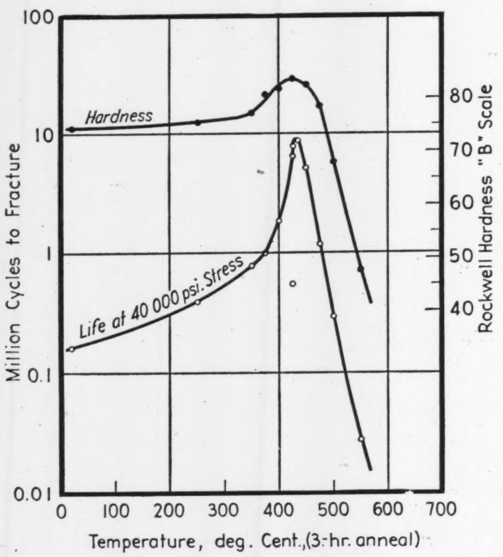

FIG. 6.—Effect of Heat Treating Temperature on Hardness and Endurance of Chromium Copper (3326).

Acknowledgments:

The work described in this paper was performed in the Technical Dept. of the American Brass Co. under the general direction of J. R. Freeman, Jr., Technical Manager and, prior to 1938, of the late H. C. Jennison. R. W. Van Wagner assisted in performing the tension tests and E. W. Palmer is responsible for many of the details of the machine design.

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(12) Discussion by A. R. Anderson of paper by C. S. Smith, "Proportional Limit Tests on Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 877 (1940).

DISCUSSION

Mr. H. F. Moore.[1]—In the paper on fatigue tests of some copper alloys the authors have given us some useful data on alloys whose fatigue properties hitherto have been very largely unknown. As the authors point out, their data obtained by tests of small polished specimens are to be regarded as data for endurance strength of the metal under the most favorable conditions. In actual machine parts, surface conditions, stress raisers, corrosion, and perhaps other factors may profoundly modify the fatigue strength of the part, sometimes quite overshadowing the inherent fatigue strength of the material. This does not mean that such data as this paper presents are without value; such data serve as basic data for a metal, upon which may be superimposed studies of various factors modifying fatigue strength in service.

Mr. Cyril Stanley Smith[2] (*author's closure*).—As Mr. Moore points out, it is well known that tests made on unnotched specimens with polished surfaces give values for endurance strength higher than the stresses that can safely be applied to machine parts or structural members of the same materials; moreover, the relative merit of various materials will depend on the condition of the surface of the piece. For this reason the values given in this paper obviously cannot be taken as design values and were not so intended. They do provide, however, a base value of more or less ideal endurance strength on which the effect of surface condition, stress raisers, corrosion, and other factors can later be evaluated.

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COMPARATIVE VALUE OF ARSENIC, ANTIMONY, AND PHOSPHORUS IN PREVENTING DEZINCIFICATION

By W. Lynes[1]

Synopsis

Early attempts to inhibit dezincification of alpha brasses culminated successfully in the introduction of a few hundredths of a per cent of arsenic. Later, antimony or phosphorus was used in similar amount for the same purpose. Laboratory tests are reported herein on 70-30 and admiralty brasses modified by an appropriate addition of each of the above elements. These tests indicate that dezincification of alpha brass alloys effectively may be suppressed by a small addition (for example, 0.03 per cent) of arsenic, antimony or phosphorus, without impairment of the alloy in other respects.

The tendency of the zinc-rich brasses to dezincify under certain conditions of exposure is well known to the producer and consumer of these alloys. The high zinc brasses are particularly liable to dezincification when in contact with sea water, but may eventually fail from this cause when exposed to many fresh water supplies or to certain aqueous solutions.

In some cases, increased service life will result from substitution of an alloy immune or strongly resistant to dezincification, such as copper or 85–15 brass. In other applications, the advantage thus obtained is offset by relative weakness of the substituted alloy in other respects. For example, copper or low brass tubes when exposed to corrosive sulfur compounds on the oil side of an oil-refinery condenser are less resistant than high-brass tubes. Then again, copper or low-zinc brasses are not useful for carrying flowing sea water, as in marine condensers. Even at water velocities of a few feet a second, protective surface layers of copper corrosion products developed on the surface of the tubing in service are not tenaciously adherent. The protective zinc-bearing corrosion products formed on the high brasses are dislodged mechanically less readily than are analogous "scales" produced on copper or zinc-poor brasses (**22**).[2]

Nature of Dezincification

The term "dezincification" was derived from a concept that since has been abandoned. It was thought that zinc was dissolved from the brass preferentially, leaving copper as a residue. For example, in 1898, Arnold (**2**) investigated the cause of the explosion of a brazed copper pipe and found deterioration of the brazing metal used in its manufacture. He concluded, in part, that, "Explosion was due to a gradual removal from the greater part of the brazing of about one half of its zinc, the remainder being converted to oxide, and thus leaving a spongy and hence brittle mass of metallic copper possessing little cohesion." Subsequent investigators repeatedly ascribed the type of corrosion observed by Arnold to selective corrosion of zinc. Yet, this opinion was not held universally. Tilden (**1**), as early as 1886, reported deposition of copper on brass immersed in sea water at 40 C. (105 F.). In 1909, Rhead (**4**), and, in 1915, Evans (**11**) suggested that dezincification might be the result of redeposition of copper. By 1923 it had been definitely shown that, in the so-called dezincification reaction, brass dissolves for the most part, if not wholly, as an entity, followed by redeposition of copper from solution. Thus, the essential difference between dezincification and other types of corrosion in which metal is generally or locally dissolved lies in the nature of the cathodic reaction: dezincification results when copper rather than hydrogen is the reactant at the cathode. Redeposited copper tends to accelerate subsequent corrosion by formation of the electrolytic cell, brass-electrolyte-copper.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this paper, see p. 877.

Clarification of the mechanism of dezincification was due largely to the intensive studies of Bengough and associates, and Abrams. Bengough and coworkers expressed the opinion in 1919 (**14**) that selective corrosion of brass may never occur. In the following year (**15**), the conclusion was reached definitely that so-called residual copper was in actuality redeposited.

In 1922, objections to the assumption that dezincification resulted from "leaching" of the zinc were pointed out by Abrams (**18**) on the basis of experimental work: namely, (1) the copper occurred in a distinct, separable layer instead of being continuous with the unattacked brass; (2) aeration or stirring of a normal solution of hydrochloric acid prevented dezincification, which otherwise occurred; (3) dezincification could not be produced in a solution of concentrated hydrochloric acid or saturated sodium chloride.

The work of Abrams emphasized the importance of a relatively high concentration of copper ions at the surface of the brass as a prelude to dezincification. This situation is realized best under the following environmental conditions: (1) moderate concentrations of certain salts or acids, especially chlorides; (2) a limited amount of dissolved oxygen; (3) low water velocity, particularly stagnation; and (4) elevated temperature.

When conditions over the entire metal surface are favorable for dezincification, the copper is redeposited as a layer. Conditions may arise, however, particularly as the result of nonuniform oxygen distribution, that produce dezincification only at certain areas. The corrosion pits thus formed are filled with "plugs" of redeposited copper.

Other alloys, such as high aluminum or manganese bronze, suffer analogous types of corrosion.

PREVENTION OF DEZINCIFICATION

In 1910 a monumental study of condenser-tube corrosion was begun by the Corrosion Research Committee of the (British) Institute of Metals. The general progress of the work was reported in a series of eight papers which appeared in the *Journal of the Institute of Metals* in the period 1911 to 1929. The present state of knowledge of condenser-tube corrosion is largely due to this work and to the further investigations that it stimulated.

Because of the practical importance of avoiding dezincification of condenser tubes and other brass parts for marine service, it was natural that the committee should give immediate and vigorous attention to the problem. At that time dezincification was the usual cause of failure of the brass tubes of marine condensers. The reciprocating engine then used led to comparatively low velocity and high temperature of the cooling water in the condenser—conditions favorable to dezincification. Later, concurrent use of the steam turbine in conjunction with a small, compact surface condenser necessitated higher water velocities. This change in operating conditions promoted failure of 70–30 brass or admiralty metal tubes by erosion-corrosion rather than dezincification.

In the Second and Third Reports to the Corrosion Committee (**7**, **12**) the superior resistance to dezincification of admiralty metal (70–29–1 tin) in comparison with 70–30 brass was established.

INFLUENCE OF ARSENIC IN PREVENTING DEZINCIFICATION

Brief mention of the remarkable efficacy of a few hundredths of a per cent of arsenic in suppressing dezincification is contained in a communicated discussion of Abrams' paper by Bengough and May (**19**). Following this suggestion, Nixon (**23**), in 1924, tested brass containing "less than 0.3 per cent of arsenic" as follows: 0.5 N NaCl, 0.5 N HCl solution for 85 days; 0.5 N $CuSO_4$, 0.5 N H_2SO_4, 29 days; acidified $CuSO_4$, 32 days; and sea water, as anode, 12 days. Dezincification did not occur. Nixon commented that "it is evident that arsenic plays a remarkable part here, as it does in many other cases .... From the above experiments it is apparent that a small fraction of 1 per cent of arsenic in brass will greatly retard, if not completely prevent, dezincification."

In the same year, Bengough and May (**22**) presented the Seventh Report to the Corrosion Committee of the Institute of Metals, concerned mainly with the mode of formation and behavior of "scales" which form on copper and brass in sea water, but chiefly remembered in relation to the work on inhibition of dezincification. For periods up to 16 weeks 70–30 brass containing 0.01 to 0.04 per cent arsenic was immersed in sea water at 50 C. (120 F.). Dezincification was absent when more than 0.01 per cent arsenic was included. "In view of the remarkable effect of arsenic in influencing the nature of the corrosion products of brass, and through them the rate of corrosion of the alloy itself, it was decided to examine the influence of a number of the metals which commonly occur in brass, either as accidental or intentional additions." Pure 70–30 brass was modified by: 1 per cent tin, 0.5 to 1 per cent nickel, 0.5 to 0.75 per cent aluminum, 0.5 per cent tungsten, 0.04 to 2 per cent manganese, 0.5 to 1 per cent iron. These alloys were tested in the same manner as the arsenical alloy. Tin, nickel, tungsten, and aluminum all had an effect of the same kind as arsenic, but much less intense, and in the order named. Manganese and iron increased the rate of dezincification under the conditions of test.

Recently, May (**26**), summing up practical experience, commented that the tendency of aluminum brass to undergo dezincification "is completely removed by a suitable small addition of arsenic (for example, 0.02 per cent), without in any way affecting the resistance of the alloy to impingement attack."

Although the value of arsenic in preventing dezincification of alpha brasses is now thoroughly established, its effectiveness in alpha-beta brasses, such as Muntz metal (60 copper, 40 zinc) is questionable. Recorded information is meager. Masing (24) noted no difference in the dezincification resistance of Muntz metal with or without 0.05 per cent arsenic after eight days in 3 per cent sodium chloride plus 0.10 per cent nitric acid solution at 50 C. (120 F.). Under the same conditions, 70–30 brass containing 0.02 to 0.05 per cent of arsenic did not dezincify. Whether more ample additions of arsenic (for example, 0.50 per cent), or the use of some other element or combination of elements, would inhibit or substantially reduce dezincification of alpha-beta brass is a matter for future investigation.

EFFECT OF ANTIMONY AND OF PHOSPHORUS

Inasmuch as arsenic, antimony, and phosphorus occur in the same group in the periodic table of the elements, reasonably it might be expected that their influences on dezincification would be similar. As regards antimony, experiments recorded in the literature are contradictory. Nixon (23) reported that, "a brass containing less than 0.5 per cent of antimony was tested, but found to be of no value. Antimony in this quantity has practically no influence upon dezincification." Experimental details were not given.

Subsequent published reference to antimony apparently does not occur until 15 yr. later (1939) when Mitchell (29) claimed "that the addition of antimony in small quantities to brasses, including aluminum brass and admiralty, definitely is beneficial to their corrosion-resistance to water. As small an amount as 0.02 per cent prevents dezincification of 65–35 alpha brass. Even as small an addition as 0.007 per cent antimony greatly increases the corrosion resistance of the brass to water, although superficial dezincification still occurs. The effect of antimony on the dezincification of brasses is greater than that of arsenic and, what is even more important, intercrystalline corrosion[3] does not occur." Conditions of test were: water-line test in "dilute synthetic sea water" at 60 C. (140 F.) for 100 days; water-line test in $\frac{1}{2}$ per cent malic acid plus $\frac{1}{2}$ per cent sodium chloride at 60 C. (140 F.) for 30 days.

Recently, phosphorus has been proposed as a dezincification inhibitor. In substantiation of the claim, Gleason (31) has briefly presented the results of laboratory tests. Tension specimens of phosphorized and of plain admiralty metal were immersed for 60 weeks in 1 per cent cupric chloride solution at 80 F. and at 120 F. Curves were drawn showing the relationship between tensile strength and elongation of the alloys, and time of immersion. The amount of phosphorus added was not mentioned, nor was it stated whether any dezincification of the phosphorized admiralty occurred.

Most recently, Barry (32) compared the corrosion resistance of plain admiralty metal with that containing 0.03 and 0.06 per cent arsenic, 0.05 per cent antimony, and 0.03 per cent phosphorus. The method of test was identical with that of Gleason. In general, the results substantiate those of Gleason and of Mitchell.

PROCESS OF INHIBITION

The precise manner in which arsenic, antimony, or phosphorus prevents or delays dezincification is not definitely known. In reference to the influence of arsenic, various theories have been offered. Bengough and May suggested, tentatively, several hypotheses: (1) "that it [arsenic] acts as a carrier of oxygen to the brass-surface and oxidizes up to a higher state the first formed copper corrosion products," (2) "that it increases the discharge potential of copper on brass," and (3) "that it tends to precipitate cuprous chloride from colloidal suspension in chloride solutions."

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[3] Intercrystalline corrosion will be considered in Part II of this paper.

Masing (24) believed that: a coating of elementary arsenic forms on the surface of corroding arsenical brass; this arsenic film being of nobler potential than the brass serves as cathode; the cathodic overvoltage of the arsenic is less than that of brass, hence depolarization of hydrogen is more readily accomplished, so readily in fact that the necessary potential for the precipitation of copper cannot be obtained. Masing also suggested that the inability of arsenic to prevent dezincification of alpha-beta brass was related to the less noble potential of the beta constituent, which favored copper redeposition at the cathode. He further suggested the probability that dezincification of alpha-beta brass could be prevented by sufficient reduction of its cathodic overvoltage, for example, by addition of an impurity more noble than arsenic.

Masing determined experimentally that the cathodic overvoltage of arsenic or arsenical brass in normal hydrochloric acid solution is approximately 0.45 v., and that of brass or antimonial brass (0.10 per cent antimony) under the same conditions, 0.56 v. Masing comments that "the fact that the antimony content is without effect on the cathodic reaction of the brass is probably explained by the fact that antimony is less noble than arsenic and is not precipitated by pickling. It also could not prevent the dezincification of alpha brass." Thus, the theory of Masing fails if antimony is effective in inhibiting dezincification.

Whereas Masing believed that the effect of arsenic found its full explanation in its influence on the cathodic reaction, Fink (28) hypothesized that arsenic, while undoubtedly having some effect at the cathode, acts chiefly as an anodic inhibitor.

On the basis of admittedly insufficient experimental evidence, Fink suggested that, in the absence of arsenic, the first stage of dezincification consists of selective corrosion of zinc at zinc-rich points, leaving residual copper spots which act as cathodes. Then, on the main part of the surface, copper and zinc are dissolved simultaneously, and the copper is redeposited on the cathodic nuclei. Arsenic brass was pictured as developing an arsenic-bearing film preferentially over those susceptible anodic spots which, otherwise, would tend to lose zinc. This film presumably acts as a barrier to selective corrosion, while permitting general corrosion of more homogeneous areas.

Most recently Hollomon and Wulff (25) have examined, by X-ray and electron diffraction methods, surface films developed by corrosion of arsenical copper, arsenical 70-30 brass, and antimonial 70-30 brass. The authors conclude, in part, that, for brass containing around 0.03 per cent or more of arsenic, arsenic redeposits and is oxidized to arsenious oxide (As_2O_3). For the antimonial brass, the mechanism of deposition is essentially similar; yet redeposited antimony does not oxidize as readily.

EXPERIMENTAL WORK

The degree of resistance to dezincification and rate of corrosion of 70-30 brass and admiralty metal, with and without small additions of arsenic, antimony, or phosphorus were compared by means of laboratory tests.

Material and Treatment:

Brasses of the analyses given in Table I were cast from cathode copper, high-grade zinc, Straits tin, 15 per cent arsenic copper, antimony, and 15 per cent phosphorus copper. Fifty-pound charges were melted in a graphite crucible, under charcoal, using a gas-fired laboratory furnace. The heats were cast in a 3-in. diameter cast iron mold. The castings were hot rolled to $\frac{7}{8}$ in. in diameter, then successively pickled, cold drawn, and annealed, according to usual commercial practice, to 0.350-in. diameter rod. After a final reduction of 50 per cent in area, the rod was finish annealed in a laboratory electric heat-treating furnace. The grain size was controlled within the range 0.045 to 0.065 mm. in diameter. The same material was used for the tests described in Part II of this paper.

TABLE I.—CHEMICAL COMPOSITION IN PER CENT OF BRASS ALLOYS USED FOR TESTS.

Nominal Composition	Copper	Tin	Arsenic	Antimony	Phosphorus	Zinc, by difference
Plain 70-30 brass	70.24	....	....	....	....	29.76
70-30, 0.03 As	71.17	....	0.039	....	....	28.79
70-30, 0.03 Sb	70.28	....	....	0.030	....	29.69
70-30, 0.03 P	71.21	....	....	....	0.036	28.75
70-30, 0.10 As	71.60	....	0.097	....	....	28.30
70-30, 0.10 Sb	70.00	....	....	0.098	....	29.90
70-30, 0.10 P	71.24	....	....	....	0.106	28.65
Plain admiralty	70.88	1.07	....	....	....	28.05
Admiralty, 0.03 As	70.11	1.08	0.030	....	....	28.78
Admiralty, 0.03 Sb	71.04	1.07	....	0.033	....	27.86
Admiralty, 0.03 P	70.25	1.05	....	....	0.034	28.67

Corrosion Tests:

The observed liability of many brasses to dezincify in sea water early inspired the use of sodium chloride or other chloride compounds in laboratory corrosion tests. Of particular importance, correlation has been obtained between such tests and service performance. Since previous work at this laboratory had demonstrated the suitability of the 5 per cent cupric chloride, 5 per cent hydrochloric acid solution for test purposes, solutions of this composition were used. Two series of tests were run: one at 72 F. ± 4 F.; the other at 150 F. ± 3 F.

Test specimens were in the form of unmachined tension specimens $7\frac{1}{2}$ in. long. According to established corrosion-testing practice, the progress of the corrosion was evaluated by comparing the tensile strengths and elongations of the samples at intervals of time during the test with the same properties before test. In conjunction with this, the surfaces of all specimens periodically were inspected visually; and, at the completion of the test, microsections were prepared and examined from duplicate samples of each alloy.

Details of the test are as follows: for each alloy, ten test specimens, annealed to 0.045 to 0.065 mm. in grain diameter and pickled, were cleaned in ether and set in holes drilled equidistant circumferentially in a 4-in. diameter by $\frac{1}{4}$-in. bakelite disk. The assembly was completely immersed in the test solution, contained in a 3-liter beaker, and the beaker covered. In the case of the tests run at 150 F. ± 3 F., the beakers were placed in an insulated container, electrically heated, with rheostat control.

All tests were run for ten weeks. Slight losses of solution by evaporation were compensated periodically. At intervals of two weeks, for the first six weeks, and after a final interval of four weeks, duplicate specimens of each alloy were removed from the solutions for mechanical tests. The surfaces of all specimens were inspected visually at these times. At the end of ten weeks, two specimens of each alloy were removed for microexamination. At the end of the second, fourth, and sixth weeks, all solutions were renewed.

Surfaces and Microstructures of Corroded Alloys:

At the end of two weeks, the plain 70-30 and plain admiralty brasses showed surface-redeposited copper. In the case of the admiralty metal immersed in solution at 72 F., the copper layer was superficial and not everywhere present. For the same alloy at 150 F., the surfaces of the specimens were completely covered by a layer of copper of appreciable depth.

The surfaces of the inhibited alloys, on the other hand, in both "cold" and "hot" solutions, contained no redeposited copper, but rather a thin adherent black deposit, probably composed of or containing the inhibiting element. In addition, the admiralty metal showed a thin, white, superimposed coating presumably tin oxide. All alloys carried a small amount of green copper oxychloride. In so far as could be judged from visual examination, the corrosion products as described above did not change in character over the period of ten weeks, nor was there material alteration in amount, except for increasing thickness of the copper layer on the uninhibited alloys. Plain admiralty in the solution at room temperature was an exception to this generalization: here, the redeposited copper consistently was of superficial thickness.

TABLE II.—AVERAGE TENSILE STRENGTHS AND ELONGATIONS OF VARIOUS 70-30 AND ADMIRALTY BRASSES, IMMERSED IN 5 PER CENT CUPRIC CHLORIDE—5 PER CENT HYDROCHLORIC ACID.

Nominal Composition	Solution at 72 F. for Periods up to Ten Weeks					Solution at 150 F. for Periods up to Ten Weeks				
	Original	Second Week	Fourth Week	Sixth Week	Tenth Week	Original	Second Week	Fourth Week	Sixth Week	Tenth Week
AVERAGE TENSILE STRENGTH, PSI.										
Plain 70-30 brass	46 500	44 500	42 300	37 300	22 200	46 500	39 500	36 400	23 700	2 200
70-30, 0.03 As	48 000	48 200	48 000	45 000	43 500	48 000	47 800	45 000	40 600	29 500
70-30, 0.03 Sb	46 800	46 700	46 500	44 800	43 000	46 800	46 000	43 000	35 200	24 100
70-30, 0.03 P	51 300	51 500	51 300	48 500	46 000	51 300	50 500	47 300	42 200	30 300
70-30, 0.10 As	47 000	46 800	46 500	43 800	41 300	47 000	45 300	43 300	37 400	26 500
70-30, 0.10 Sb	46 500	46 300	45 300	42 300	40 500	46 500	45 500	41 800	36 800	26 800
70-30, 0.10 P	51 300	51 000	50 500	47 300	45 000	51 300	50 500	46 800	39 500	28 200
Plain admiralty	50 800	50 800	50 500	46 300	44 000	50 800	45 800	32 800	25 500	2 400
Admiralty, 0.03 As	47 800	48 000	48 000	45 800	43 000	47 800	47 800	42 300	37 000	26 700
Admiralty, 0.03 Sb	50 500	50 200	50 300	47 800	44 800	50 500	50 300	46 300	40 300	28 000
Admiralty, 0.03 P	53 500	53 600	53 000	49 800	45 800	53 500	51 800	48 500	39 300	27 900
AVERAGE ELONGATION, PER CENT IN 1.4 IN.										
Plain 70-30 brass	80	80	73	57	39	80	66	59	44	..
70-30, 0.03 As	77	73	71	68	67	77	71	70	64	60
70-30, 0.03 Sb	77	76	75	67	69	77	75	68	63	53
70-30, 0.03 P	74	73	70	64	61	74	74	68	61	67
70-30, 0.10 As	78	76	70	73	72	78	78	71	69	63
70-30, 0.10 Sb	77	75	74	71	69	77	73	70	67	64
70-30, 0.10 P	71	69	70	65	66	71	69	68	66	66
Plain admiralty	75	80	74	72	73	75	68	47	46	..
Admiralty, 0.03 As	85	79	79	73	73	85	82	75	71	66
Admiralty, 0.03 Sb	79	77	74	74	70	79	72	72	73	66
Admiralty, 0.03 P	69	70	67	63	63	69	67	62	61	56

The specimens taken at the end of the test for microexamination were, in each case, cross-sectioned on several planes. The sections were mounted in transparent plastic, and polished. In regard to the uninhibited alloys: specimens of both 70-30 and admiralty brass tested at 150 F. were on the verge of complete dezincification (see Fig. 1). By repeated cross-sectioning, it was established that each alloy contained a central core of unattacked brass about 0.05 in. in diameter along the greater part of the axis of the specimen. However, occasional planes were obtained on which the brass had been completely replaced by copper. For the same alloys tested at room temperature, the situation, quantitatively, was quite different. As previously stated, but a slight amount of copper had been redeposited on the admiralty metal. The dezincified zone of the 70-30 brass was about 0.05 in. in thickness.

TABLE III.—AVERAGE PER CENT LOSSES IN TENSILE STRENGTHS OF VARIOUS 70-30 AND ADMIRALTY BRASSES, IMMERSED IN 5 PER CENT CUPRIC CHLORIDE—5 PER CENT HYDROCHLORIC ACID SOLUTION AT 72 F., AND AT 150 F. FOR PERIODS UP TO TEN WEEKS.

Nominal Composition	72 F.				10 F.			
	Second Week	Fourth Week	Sixth Week	Tenth Week	Second Week	Fourth Week	Sixth Week	Tenth Week
Plain 70-30 brass	4.3	9.1	19.8	52.3	15.0	21.8	49.0	95.3
70-30, 0.03 As	0.0	0.0	6.2	9.4	0.4	6.3	15.4	38.5
70-30, 0.03 Sb	0.2	0.6	4.3	8.2	1.7	8.1	25.0	48.5
70-30, 0.03 P	0.0	0.0	5.5	10.3	1.6	7.8	17.7	41.0
70-30, 0.10 As	0.4	1.1	6.8	12.1	3.6	7.9	20.5	43.6
70-30, 0.10 Sb	0.4	2.6	9.0	12.9	2.1	10.1	20.8	42.5
70-30, 0.10 P	0.6	1.6	7.8	12.3	1.6	8.8	23.0	45.0
Plain admiralty	0.0	0.6	8.5	13.4	9.9	35.4	50.3	95.2
Admiralty, 0.03 As.	0.0	0.0	4.2	10.1	0.0	11.5	22.7	44.2
Admiralty, 0.03 Sb.	0.6	0.4	5.4	11.2	2.4	8.5	20.2	44.5
Admiralty, 0.03 P.	0.0	0.9	6.9	14.4	3.2	9.4	26.6	47.8

The alloys containing arsenic, antimony or phosphorus were examined carefully up to a magnification of about 500 for the possible presence of intergranular corrosion. Intergranular corrosion could not be detected (see Fig. 2).

MECHANICAL TESTS

Tension tests were made with a Southwark-Emery hydraulic testing machine. Tensile strengths were computed on the basis of the original cross-sectional areas, and elongations measured over a gage length of four times the diameter (1.4 in.). Results of the tests are given in Tables II and III.

SUMMARY AND DISCUSSION

Conditions were established in the laboratory that resulted in complete dezincification of 0.350-in. diameter plain 70-30 brass and admiralty metal rod in a period of ten weeks. Under these conditions, the same alloys modified by about 0.03 per cent arsenic, antimony, or phosphorus did not dezincify. Neither did corrosion of the modified alloys progress along grain boundaries. Wholly, the corrosion resulted in approximately uniform reductions of diameter of the specimens. Quantitative indices of corrosion rates are given in Tables II and III.

The test results on the uninhibited admiralty alloy are of interest. It is well established, through laboratory tests confirmed by service experience, that the 1 per cent of tin contained in admiralty metal usually is a retardant of dezincification. The results of our corrosion tests are in accord, when temperatures of around 72 F. are involved. But, at 150 F., tin was without influence on the rate of the dezincification reaction. Increase of temperature from 72 F. to 150 F. was markedly and similarly accelerative of dezincification for both the admiralty and the 70-30 brass. Not unexpectedly, the following generalization appears to be warranted: under conditions where dezincification may occur, the higher the temperature of usage, the more urgent the need of an effective inhibitor in alloys susceptible to dezincification.

PART II—INFLUENCES OF THE INHIBITOR

The second part of this paper comprises a consideration of possible effects of the inhibitor aside from suppression of dezincification. More particularly, valid evidence has been sought that would indicate an adverse influence on the performance of the alloy as the result of the use of any specific inhibitor.

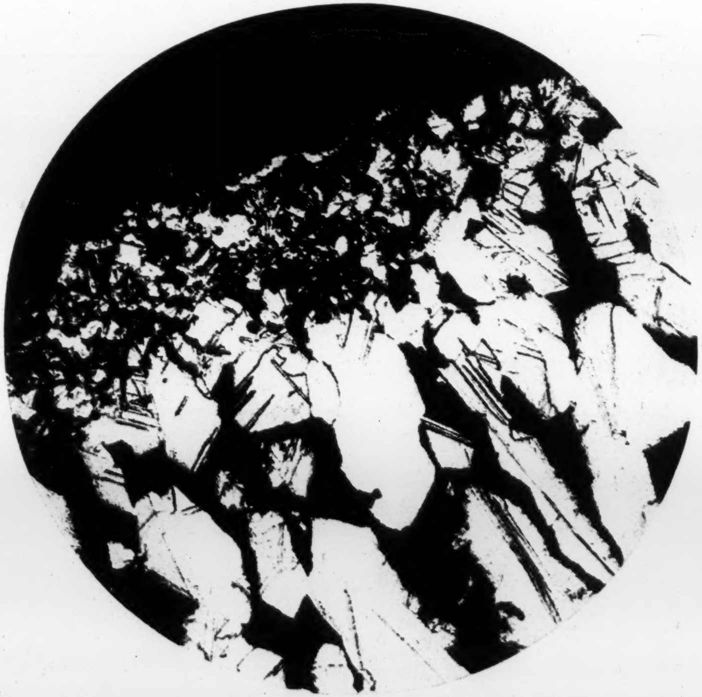

FIG. 1.—Cross-Section Through Edge of Plain Admiralty Metal Corrosion-Test Specimen After Total Immersion for Ten Weeks in 5 per cent $CuCl_2$ - 5 per cent HCl solution at 150 F. (65 C.). Shows complete replacement of brass by redeposited copper (× 75).

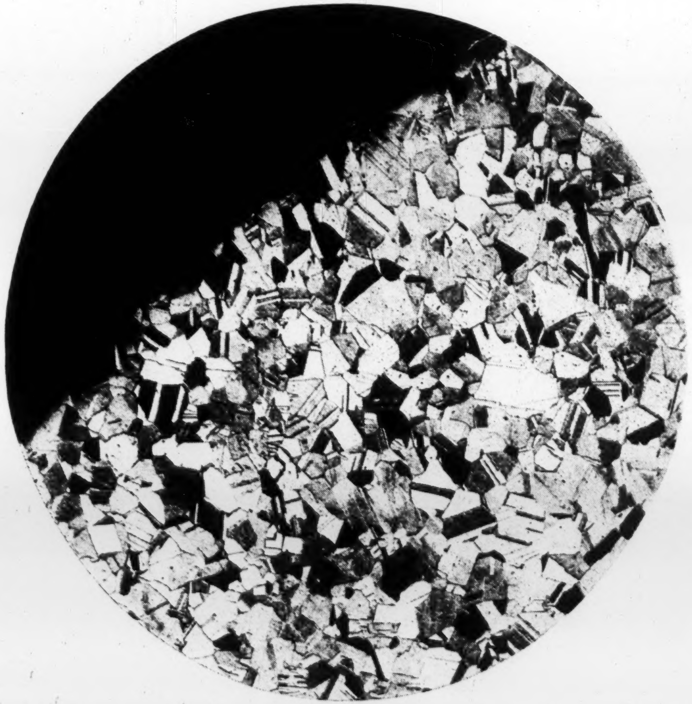

FIG. 2.—Same Location and Test Conditions as in Fig. 1. Admiralty metal containing 0.04 per cent arsenic shows absence of redeposited copper or intergranular corrosion (× 75).

For, it is well to consider whether the addition of an element to control dezincification may, under some conditions, decrease rather than increase the life of the fabricated part.

In general, the presence of an inhibitor could be damaging to the performance of a brass part, such as a tube, were it able to impair its chemical or mechanical resistance to its environment. Before considering in detail whether the possibility of such damage exists, it is convenient to tabulate the types of corrosion and the mechanical properties of the alloy that significantly might be influenced by the addition of arsenic, antimony, or phosphorus in the amount commercially used:

- (*A*) Chemical Influence
  - 1. Uniform corrosion (general thinning)
  - 2. Nonuniform corrosion
    - (*a*) Pitting and grooving
    - (*b*) Intergranular corrosion
- (*B*) Chemical-Mechanical Influence
  - 1. Stress-corrosion
    - (*a*) Intergranular cracking
    - (*b*) Corrosion-fatigue
  - 2. Erosion-corrosion
- (*C*) Mechanical Influence
  - 1. Tensile strength and elongation
  - 2. Notch sensitivity
  - 3. Endurance limit

(*A*) CHEMICAL INFLUENCE

1. *Uniform Corrosion (general thinning):*

Conditions may exist that cause brasses such as 70-30 or admiralty to corrode uniformly without dezincifying. When no tendency exists for copper to redeposit from solution, the presence of an inhibitor conceivably might accelerate corrosion. Laboratory corrosion tests designed to evaluate this possibility or observations based on practical experience were not found recorded in the literature. Tests made at this laboratory on uninhibited and inhibited admiralty are described below. Under the test conditions, the rate and degree of uniformity of corrosion of all alloys were substantially the same. Consistently, however, the rate of corrosion of the plain admiralty was slightly higher than the rate of the same alloy modified by arsenic, antimony, or phosphorus.

TABLE IV.—AVERAGE TENSILE STRENGTH, ELONGATION, AND LOSS IN TENSILE STRENGTH OF VARIOUS ADMIRALTY ALLOYS IMMERSED IN AERATED 10 PER CENT HYDROCHLORIC ACID SOLUTION AT 72 F. FOR PERIODS UP TO 8 WEEKS.

Nominal Composition	Original	Second Week	Fourth Week	Sixth Week	Eighth Week
AVERAGE TENSILE STRENGTH, PSI.					
Plain admiralty	50 800	44 500	36 000	24 200	12 000
Admiralty, 0.03 As	47 800	44 800	36 300	26 300	14 700
Admiralty, 0.03 Sb	50 500	45 300	38 700	28 000	14 100
Admiralty, 0.03 P	53 500	48 400	39 300	28 100	15 600
AVERAGE ELONGATION IN 1.4 IN., PER CENT					
Plain admiralty	75	77	73	68	59
Admiralty, 0.03 As	85	80	80	74	67
Admiralty, 0.03 Sb	79	74	70	69	58
Admiralty, 0.03 P	69	66	64	53	48
LOSS IN TENSILE STRENGTH, PER CENT					
Plain admiralty	...	12.4	29.2	52.4	76.4
Admiralty, 0.03 As	...	6.3	24.1	45.0	69.3
Admiralty, 0.03 Sb	...	10.3	23.4	44.5	72.1
Admiralty, 0.03 P	...	9.5	26.6	47.4	70.9

Experiments.—The admiralty alloys listed in Table I were used for test. The preparation of specimens and method of test were the same as those described in Part I of this paper,[4] except for these differences: the solution consisted of 10 per cent hydrochloric acid in water at room temperature. Compressed air was introduced through a glass tube, centrally at the bottom of each beaker, at a rate of about one-half liter per minute. The purpose of the air was to increase oxidation of dissolved copper to insoluble copper oxychloride, and thereby to maintain the concentration of copper ions in solution below that necessary for redeposition. The test was run for eight weeks, with removal of samples at the end of every two weeks.

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[4] See pp. 863-866.

The appearance of the surfaces of the corroded, inhibited alloys, and the results of their microexamination at the end of the test, were essentially the same as described in Part I. Contrary to the results of the previous tests, and by intention, copper did not redeposit on the plain admiralty. The surfaces of these specimens were practically free from corrosion products, except for a little copper oxychloride.

Results of mechanical tests are given in Table IV.

2. *Nonuniform Corrosion:*

(*a*) *Pitting and Grooving.*—The influence of an inhibitor on the corrosion rate of uniformly dissolving brass has just been considered. An analogous consideration, even more important from a practical viewpoint, is the influence of the inhibitor on the rate of and tendency toward pitting, when dezincification does not occur.

May (26), after commenting on the complete inhibition of dezincification by the addition of 0.02 per cent arsenic to aluminum brass, adds: "An even higher arsenic content may be used if the tubes are required especially to resist deposit attack; an aluminum brass with 0.04 per cent or 0.05 per cent arsenic resists deposits of sand far better than any known condenser-tube alloy, with the possible exception of those alloys containing a very high percentage of nickel." As is well known, electrically nonconductive deposits, such as sand, wood, shells, etc., promote corrosive attack by producing oxygen-concentration cells. Therefore, the observation that addition of arsenic to aluminum brass increases its resistance to deposit attack is equivalent to the statement that arsenic decreases differential aeration currents. The practical importance of this observation is evident. Oxygen concentration cells are an important cause of rapid pitting corrosion and are produced not only by deposits but also form readily in crevices, at a water line, in drops of liquid, and in porous "scale" or sediment.

That arsenic diminishes the strength of currents set up in a differential aeration cell has been observed by Fink (28) by means of potential measurements on arsenical and nonarsenical 70–30 brass.

(*b*) *Intergranular Corrosion.* — Recently Mitchell (29) and Barry (32) have reported intergranular corrosion of arsenical admiralty metal under laboratory tests. Intergranular attack was said to have occurred in the absence of appreciable stress. This type of intergranular penetration must not be confused with the well-known "season cracking" of brass, which likewise follows an intergranular path, but requires simultaneous action of stress and corrosion.

The test results of Mitchell and Barry are of considerable scientific interest; for, in so far as we know, this is the first time that report has been made of intergranular corrosion of a copper-base alloy in the absence of internal or external stress of appreciable magnitude.

Mitchell's test conditions were: a water-line test in "dilute synthetic sea water" at 60 C. (140 F.) for 100 days. The alloys tested were: admiralty metal, aluminum brass, and leaded 2 and 1 brass, in three conditions —plain, with antimony, and with arsenic. The fabricating procedure or final treatment of the alloys was not given. Corrosion resistance was evaluated on the basis of percentage loss of original tensile strength. Intergranular corrosion was noted only in the case of arsenical admiralty (0.06 per cent arsenic). Barry tested five "finish-annealed" admiralty metal alloys (plain, 0.03 per cent arsenic, 0.06 per cent arsenic, 0.05 per cent antimony, and 0.03 per cent phosphorus) by total immersion in 1 per cent cupric chloride solution at 80 and 120 F. for 50 weeks. In the tests at elevated temperature, the two arsenical admiralty samples "showed definite intercrystalline attack of the metal."

Mitchell and Barry infer that arsenic is a potentially dangerous inhibitor, responsible for service failures. This conclusion appears to be unsubstantiated, either in the literature or by practical experience.

Furthermore, the emphasis placed by the above-mentioned investigators on cracking of admiralty tubes appears to be disproportionate. Cracking is not a major cause of tube failure. In the experience of the present author, the cracks usually follow a transgranular path (corrosion-fatigue), hence cannot be related, even remotely, to intergranular corrosion.

For example, an analysis was made of the causes of failure of 46 admiralty heat-exchanger tubes from diverse sources, examined at this laboratory in the past 3 yr. Arsenic in commercial amount was contained in 23 per cent of the tubes; the remainder contained no inhibitor. Twenty-two per cent of the tubes were cracked; 15 per cent as a result of corrosion-fatigue, and 7 per cent from stress corrosion, none, apparently, from intergranular corrosion.

The lack of correlation between the corrosion behavior of arsenical admiralty in service and under specific, but unrelated, conditions in the laboratory is not surprising. Engineers have recognized for years the danger of indiscriminate use of short-time tests and the careless or misleading application of the test data, even when the tests bear some relationship to actual service conditions. Laboratory corrosion testing suffers this fundamental limitation: it is usually impossible or impractical to reproduce the conditions that the alloy must withstand in service. For this reason, such a test may be wholly unreliable in predicting type or rate of corrosion. A laboratory test is of proven value only when the results obtained thereby are capable of consistent correlation with the performance obtained under conditions of usage. That the tests described by Mitchell and Barry are of such a nature remains to be demonstrated.

The well-known limitations of laboratory corrosion tests as well as their value have been fully recognized and aptly stated by Dix (33). He has remarked in relation to stress-corrosion cracking that, "by the ingenious selection of the test conditions, it is probable that most metals in engineering use today can be made to fail by stress corrosion. To obtain results of practical value, the test conditions must be selected with due regard to the service to which the metal will be subjected. Correlation between laboratory stress-corrosion tests and service performance is difficult to obtain and often requires many years. If the test conditions are intelligently selected, accelerated stress-corrosion tests are useful in the development of new alloys. The results of such tests on new alloy compositions may be compared with those obtained on established alloys whose performance over a number of years of service is known. In this way, a comparative rating may be obtained and the probability of the new alloy failing in service by stress-corrosion estimated with considerable accuracy."

(B) CHEMICAL - MECHANICAL INFLUENCE

1. *Stress-Corrosion:*

(*a*) *Intergranular Cracking.*—Many metals and alloys fail from intergranular stress-corrosion cracking under suitable combinations of static stress, corrosion, and temperature. Among these are: various steels (**34–36**), aluminum and alloys (**37–38**), nickel-copper alloys, and various brasses and bronzes (especially the higher zinc brasses, aluminum brass and bronze, and silicon bronze) (**39–46**). These materials differ considerably in susceptibility in this sense: that common commercial usage either does or does not expose the metal frequently to an environment of the chemical and physical characteristics inducive to this type of attack. The requisite environmental conditions vary from alloy to alloy, but the corrosive attack when produced is characterized universally by selective pitting and grooving of the grain boundaries. Stated electrochemically, the grain boundary is anodic to the grain.

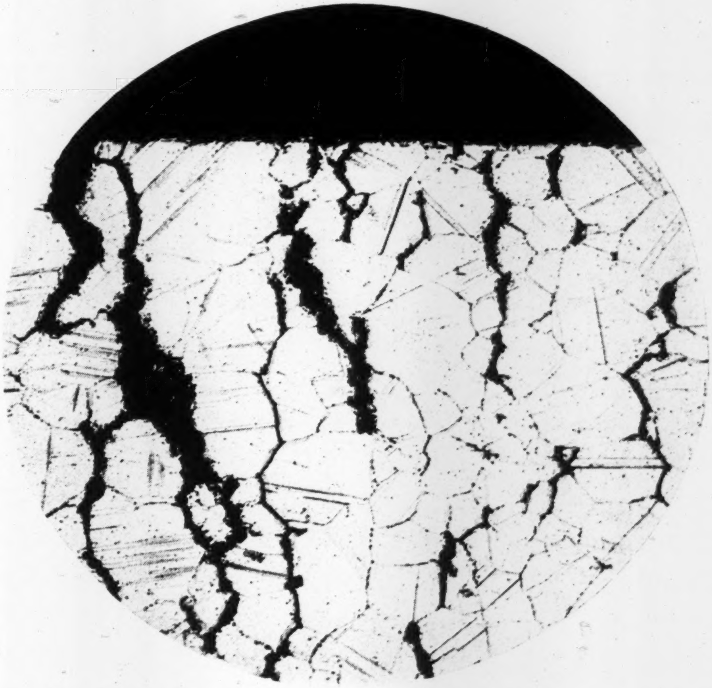

FIG. 3.—Unhibited Admiralty Metal Heater Tube. Cross-section through tube-wall at flattened U-bend. Shows intergranular stress-corrosion cracking.

The notch effect of grain boundary corrosion together with pressure developed by any corrosion products lodged therein eventually superimpose a damaging stress concentration on the stresses existing previously. Thereupon cracks are propagated along the grain boundaries, as shown in Fig. 3. This type of attack on brasses and bronzes inaptly is called "season cracking." Its occurrence in stressed brasses or bronzes is favored particularly by contact with aqueous solutions of ammonia or compounds of ammonia or mercury, or by mercury itself. The season cracking of stressed brass or bronze on atmospheric exposure depends on slight concentrations of ammonia or other accelerants in the air and the presence of moisture at the surface of the metal. Under these feebly corroding conditions, the period required to produce failure is sometimes measurable in terms of months or years; hence, the term "season cracking." When this terminology was proposed, the influence of corrosion had not been recognized; but, in view of present knowledge, "stress-corrosion" more completely would define the phenomenon.

Stresses in the material (which, in conjunction with grain boundary corrosion, cause this type of failure), may be produced by: nonuniform deformation of the alloy by cold working, by a temperature gradient during cooling from a high temperature, or by structural changes involving localized change of volume. Operating stresses usually are insufficient, except as they may be additive to internal stresses produced by the means outlined above.

Stress-corrosion (or season) cracking is an uncommon cause of failure of brass parts, such as heat-exchanger tubing. This is due largely, if not entirely, to the common practice of annealing susceptible alloys subsequent to the final cold-working operation (41).

It is pertinent to inquire whether the presence of an inhibitor in brass might favor increased susceptibility to corrosion along grain boundaries, and thus promote intergranular cracking. From the experiments described below, this possibility appears to be remote. In the case of arsenical admiralty and arsenical aluminum brass, the experimental evidence is substantiated by extended service experience.

Experiments.—The use of a mercury salt for testing in the laboratory the liability of brass to "season crack" was suggested as early as 1905 (41). This test, has given useful correlation with results in service. Because of its proven usefulness, a modification of this test was used to evaluate the influence of inhibitors on susceptibility to intergranular cracking. The method was partly on the lines of that described by Genders (45).

The material used was the same as that previously described. Inasmuch as stressless intergranular corrosion of arsenical admiralty has been said to occur under certain specific conditions of laboratory testing,[5] preliminary tests were made on the annealed material in the absence of applied stress. Duplicate 10-in. lengths of each alloy were wholly immersed in standard mercurous nitrate solution (10 per cent $HgNO_3$ plus 1.3 per cent concentrated HNO_3) for 15 min. The deposited mercury was then volatilized by means of a hot plate and tensile strengths and percentage elongations in 1.4 in. determined. The correspondence of these properties with the same properties when mercury was

TABLE V.—AVERAGE TENSILE STRENGTHS AND ELONGATIONS OF VARIOUS ALLOYS.

Nominal Composition	Average Tensile Strength, psi.		Ratio, A/B	Average Elongation in 1.4 in., per cent		Ratio, C/D
	A[a]	B[b]		C[a]	D[b]	
Plain 70-30 brass	46 300	46 500	0.99	81	80	1.01
70-30, 0.03 As	49 000	48 000	1.02	75	77	0.97
70-30, 0.03 Sb	46 300	46 800	0.99	78	77	1.01
70-30, 0.03 P	50 500	51 300	0.99	76	74	1.02
70-30, 0.10 As	47 500	47 000	1.01	78	78	1.00
70-30, 0.10 Sb	47 000	46 500	1.01	77	77	1.00
70-30, 0.10 P	52 000	51 300	1.01	72	71	1.01
Plain admiralty	50 800	50 800	1.00	79	75	1.05
Admiralty, 0.03 As	47 500	47 800	0.99	86	85	1.01
Admiralty, 0.03 Sb	51 000	50 500	1.01	80	79	1.01
Admiralty, 0.03 P	53 500	53 500	1.00	67	69	0.97

[a] Tested after previous contact with mercury for 15 min., followed by mercury evaporation.

[b] Tested after no previous contact with mercury.

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[5] See pp. 869–871.

not deposited on the surfaces of the test specimens was well within experimental error. Thus, damage of the inhibited brasses by intergranular penetration of mercury is not indicated in the absence of stress. Results are shown in Table V.

The relative susceptibility to stress-corrosion of the various alloys was tested as follows: specimens in triplicate were immersed in mercurous nitrate solution as previously described, followed by immediate determinations of tensile strengths. To avoid breaking of the specimen in the grips of the tension testing machine, the grip ends were coated with paraffin before immersion in the test solution. The ratio of the average tensile strength of the mercury-coated specimens of each alloy to the average tensile strength of untreated specimens of the same alloy was taken as an index of susceptibility to stress-corrosion. A further test was made in which the stress just insufficient to cause fracture or cracking of a mercury-coated specimen, when applied for 15 min., was determined. Again, tests were made in triplicate and the ratios computed. Results are given in Table VI. It may be seen that the ratios of the inhibited alloys rather closely fall around those of the uninhibited ones, being in some cases slightly more and in others slightly less.

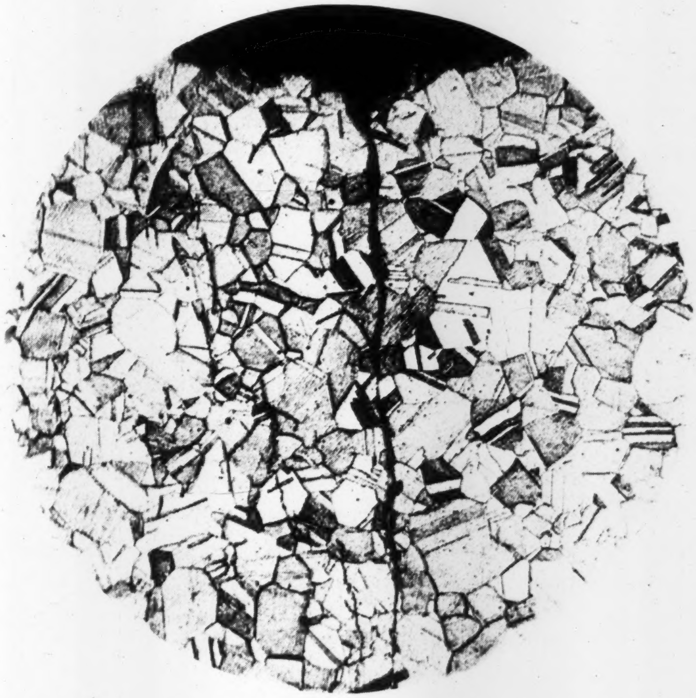

FIG. 4.—Longitudinal Section Through Tube-Wall of Plain Admiralty Metal Tube. Shows transgranular fatigue crack at base of corrosion pit (× 75).

(*b*) *Corrosion-Fatigue.*—Failure from corrosion-fatigue is not uncommon in parts subjected to the conjoint action of alternating operating stresses and corrosion. The resulting fracture is transgranular (Fig. 4), thus distinguishing the type of failure microscopically from the stress-corrosion type described above.

When corrosion and cyclic stresses act simultaneously, these influences are more than additive. A reciprocal action is involved: stress increases corrosion, and, conversely, corrosion increases stress. The alternating stresses, particularly if of high frequency and intensity, tend to disrupt or dislodge films and other corrosion products protecting the metal surface (**47**). If the accelerated corrosion thus produced is distributed uniformly, the fatigue resistance of the material will be reduced in proportion to the reduction in cross-section; but, if pitting ensues, the situation is more serious. The pits or crevices produced under conditions of corrosion and cyclic stress are characteristically narrow and pointed (**47**), a type that produces great weakening by stress-concentration effects. The final stage of the corrosion-fatigue process is the initiation of cracks at the base of these pits and their gradual propagation inward.

TABLE VI.—AVERAGE TENSILE STRENGTHS OF VARIOUS ALLOYS.

Nominal Composition	Average Tensile Strength, psi.		Ratio, A/B	Average Safe Stress, psi.	Ratio, C/B
	A[a]	B[b]		C[c]	
Plain 70-30 brass...	17 800	46 500	0.38	17 000	0.37
70-30, 0.03 As......	19 200	48 000	0.40	18 000	0.37
70-30, 0.03 Sb......	16 600	46 800	0.36	15 000	0.32
70-30, 0.03 P.......	21 900	51 300	0.43	20 000	0.39
70-30, 0.10 As......	16 400	47 000	0.35	15 000	0.32
70-30, 0.10 Sb......	15 800	46 500	0.34	14 000	0.30
70-30, 0.10 P.......	21 900	51 300	0.43	20 000	0.39
Plain admiralty....	16 500	50 800	0.32	15 000	0.30
Admiralty, 0.03 As.	16 500	47 800	0.35	15 000	0.31
Admiralty, 0.03 Sb.	16 600	50 500	0.33	15 000	0.30
Admiralty, 0.03 P...	19 100	53 500	0.36	18 000	0.34

[a] Tested in contact with mercury.

[b] Tested in contact with air.

[c] Stress just insufficient to cause cracking when applied for 15 min.

The resistance of an alloy to corrosion-fatigue depends primarily on its corrosion resistance. Thus, cold working, which increases the endurance limit of a metal in the absence of appreciable corrosion, does not have an analogous effect under conditions of corrosion-fatigue (**47**). Furthermore, cold working, particularly without subsequent relief annealing, may be deleterious because of resulting corrosion acceleration, or because of increase in notch sensitivity (**50**).

As regards the influence of an inhibitor on resistance to corrosion-fatigue, experimental work reported in this paper indicates that the endurance limit and notch sensitivity of inhibited and uninhibited alloys are comparable, and that their resistances to corrosion are similar, except under conditions promoting dezincification, where inhibited alloys are definitely superior. Therefore, increased susceptibility of a brass alloy to corrosion-fatigue as the result of an addition designed to prevent dezincification reasonably cannot be anticipated, whereas, in some cases, improvement may be expected therefrom.

2. *Erosion-Corrosion:*

The corrosiveness of a moving liquid is related intrinsically to its rate of motion past the object being corroded. As the liquid velocity increases, the formation of a corrosion-protective layer on the surface of the metal or alloy increasingly is hindered or, if formed, it is more liable to mechanical removal. In the case of heat-exchanger tubes, the velocity effect may lead to the well-known "impingement" attack (**49**). This usually occurs within a few inches of the tube sheet. If particles of solids, such as sand, or, particularly, air bubbles are present in a stream of impinging fluid, the corrosive effect may be accelerated greatly.

Inasmuch as 70–30 or admiralty brass is not, as a rule, sufficiently resistant above water velocities of around 6 ft. per sec., and is not ordinarily recommended for such usage, consideration of the effect of an inhibitor on the erosion-corrosion resistance of these alloys could serve no practical purpose. Aluminum brass and cupro-nickel tubing are characterized by excellent resistance to types of attack associated with the higher fluid velocities and have found widespread use on this basis. In regard to aluminum brass, there is no experimental evidence or practical experience to show that the resistance of the alloy is impaired by the presence of an inhibitor. May states (26) that the tendency of aluminum brass to dezincify may be "completely removed by a suitable small addition of arsenic (for example, 0.02 per cent) without in any way affecting the resistance of the alloy to impingement attack."

(C) MECHANICAL INFLUENCE

1. *Tensile Strength and Elongation:*

Bearing in mind the minute amount of an inhibitor that suffices to suppress dezincification, the possibility appears to be remote that the mechanical properties of a brass alloy would be influenced significantly by its presence. This supposition is substantiated in the case of tensile strength and elongation by the data contained in Table VII. Phosphorus appears slightly to increase tensile strength and hardness, and commensurately to decrease elongation. Arsenic or antimony are without detectable influence.

Experiments.—Material of each analysis sufficient for three tensile specimens was carefully annealed to 0.065 mm. average grain diameter. The grain size variable thus having been eliminated, differences in tensile strength and elongation beyond possible experimental error could be attributed properly to the influence of alloy modification.

2. *Notch Sensitivity:*

As already described, the formation of corrosion pits in the presence of cyclic operating stresses may result in corrosion-fatigue cracking at an applied stress value well below the endurance limit of the alloy. In this section, the possibility will be considered of damage due to corrosion notches being accentuated by the presence of an inhibitor.

In general, there are two ways in which it appears possible that the inhibitor might measurably alter sensitivity to notch effects: (1) by altering work sensitivity,[6] or (2) by an "embrittling" effect. In regard to the first, as pointed out by McAdam (50) and others, work hardening of a metal or alloy increases its notch sensitivity.[7] Fatigue stresses, (particularly when concentrated, as at the base of a notch) readily may be of sufficient magnitude to produce progressively a work-hardened layer of metal. Particularly would this be true when the initial elastic limit of the metal was low. It follows, other things being equal, that, for a given range of cyclic stresses, an alloy with greater work sensitivity would possess greater notch sensitivity.

TABLE VII.—COMPARISON OF AVERAGE TENSILE STRENGTHS AND ELONGATIONS OF VARIOUS BRASS ALLOYS AT 0.065 MM. AVERAGE GRAIN DIAMETER.

Nominal Composition	Tensile Strength, psi.	Elongation in 4X diameter, per cent	Rockwell Hardness, "F" scale
Plain 70-30 brass	46 500	80	52
70-30, 0.03 As	48 000	77	55
70-30, 0.03 Sb	46 800	77	55
70-30, 0.03 P	51 300	74	58
70-30, 0.10 As	47 000	78	54
70-30, 0.10 Sb	46 500	77	54
70-30, 0.10 P	51 300	71	59
Plain admiralty	50 800	75	60
Admiralty, 0.03 As	47 800	85	56
Admiralty, 0.03 Sb	50 500	79	60
Admiralty, 0.03 P	53 500	69	63

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[6] Percentage increase in hardness resulting from a given amount of cold working may be taken as an index of work sensitivity.

[7] Percentage decrease in fatigue limit, due to a notch is a measure of notch sensitivity under cyclic stresses.

Experiments.—The work hardening characteristics of 70–30 brass or admiralty are not altered appreciably by the addition of an inhibitor, as shown below:

Three-foot lengths of each alloy were cold drawn to 5, 10, 15, and 30 per cent reduction in area, and average Rockwell "F" scale hardnesses determined. Results are shown in Table VIII.

Notched-Rod Impact Tests.—The possibility was mentioned above of an inhibitor increasing notch sensitivity by an "embrittling" effect. To explore this possibility, notched-rod impact tests were made. It should be borne in mind that it has not been demonstrated that a connection exists between notch sensitivity as measured by a test involving continuous or impact application of load, and notch sensitivity measured by tests in which the load is cyclic, as in notched-rod fatigue testing. Therefore the results of a notched-rod impact test cannot be clearly related to alloy performance under corrosion-fatigue conditions. Yet, within the restrictions imposed by the test conditions, the test is a useful measure of relative "brittleness" in the presence of a notch. A fundamental criterion as to whether a metal breaks in a "brittle" manner, that is, with little or no plastic deformation, is the ratio of cohesive strength to resistance to deformation (51). In the case of a non-heat treatable alloy, "brittle" fracture may be favored in two important ways: (1) by cold working, which decreases the above ratio, and (2) by introduction of a stress-concentrator, such as a notch, which increases the ratio required to obtain appreciable deformation. In the experimental work, a more comprehensive picture was obtained by varying factors (1) and (2).

TABLE VIII.—RELATIONSHIP BETWEEN PERCENTAGE OF COLD REDUCTION AND ROCKWELL HARDNESS "F" SCALE FOR VARIOUS BRASS ALLOYS.

Nominal Composition	Average Rockwell "F" Scale Hardness at Various Reductions of Area				
	0	5 per cent	10 per cent	15 per cent	30 per cent
Plain 70-30 brass	51	81	87	90	98
70-30, 0.03 As	60	87	91	94	99
70-30, 0.03 Sb	63	83	88	92	98
70-30, 0.03 P	61	84	89	93	99
70-30, 0.10 As	55	83	88	91	99
70-30, 01.0 Sb	54	82	88	91	98
70-30, 0.10 P	58	86	91	94	100
Plain admiralty	57	81	88	91	100
Admiralty, 0.03 As	54	81	86	91	100
Admiralty, 0.03 Sb	58	81	87	91	99
Admiralty, 0.03 P	64	83	89	93	100

TABLE IX.—NOTCHED-ROD TESTS.

Nominal Composition	Average Impact Value After Various Reductions of Area by Cold Drawing, ft-lb.							
	0		5 per cent		10 per cent		15 per cent	
	2-mm. Notch	3-mm. Notch	2-mm. Notch	3-mm. Notch	2-mm. Notch	3-mm. Notch	2-mm. Notch	3-mm. Notch
Plain 70-30 brass	A[a]	A	A	8	15	8	11	6
70-30, 0.03 As	A	7	A	7	14	8	11	6
70-30, 0.03 Sb	A	A	A	8	16	9	13	6
70-30, 0.03 P	A	A	A	9	15	9	12	7
70-30, 0.10 As	A	A	A	8	15	9	12	7
70-30, 0.10 Sb	11	6	8	6	9	8	7	5
70-30, 0.10 P	A	A	A	9	17	10	13	8
Plain admiralty	A	A	A	8	14	8	11	7
Admiralty, 0.03 As	A	A	A	9	13	8	12	8
Admiralty, 0.03 Sb	A	9	15	8	13	8	12	8
Admiralty, 0.03 P	A	8	13	8	12	8	12	7

[a] A = incompletely fractured.

Under the conditions of the test, significant increase in notch sensitivity did not result from addition of an inhibitor to 70-30 or admiralty brass, except in the case of the alloy 70-30 brass containing 0.10 per cent antimony, which was definitely more notch sensitive. This composition, however, is outside of usual commercial limits.

Experiments.—Suitable lengths of the previously described alloys were cold drawn to 5, 10, and 15 per cent reduction in area, one length of each being retained in the annealed condition. For each condition of each alloy, two 6-in. lengths were cut. Two circumferential notches (53) with 45-deg. angles and 0.01-in. radii at the bases were machined in each length. One notch was 2 mm. deep, the other 3 mm. deep. The distance between notches was 1.1 in. These specimens were tested in an Izod impact testing machine with the results shown in Table IX.

3. *Endurance Limit:*

As in the case of properties such as tensile strength and elongation, a few hundredths of a per cent of arsenic, antimony or phosphorus would not be expected to alter measurably the endurance limit of a brass alloy. Certain small differences existed in determinations made at this laboratory. These differences are thought to result from factors other than inhibitor content and to be within possible experimental error. On the assumption that the differences are actual rather than apparent, they do not appear to be significant. For, as mentioned above,[8] the resistance of an alloy simultaneously subjected to corrosion and cycle stresses depends primarily on its corrosion resistance and to a lesser extent on its endurance limit.

Experiments.—The fatigue tests were run on R. R. Moore rotating-beam fatigue machines. Standard specimens were used, bright annealed after machining to 0.030 to 0.045-mm. grain size. The reduced sections of the specimens were polished through 000 emery paper. The endurance limit was determined at 10 million cycles. In establishing this figure, tests were run at intervals of 1000 psi. Results are shown in Table X.

TABLE X.—ENDURANCE LIMIT AT 10 MILLION CYCLES OF VARIOUS ADMIRALTY ALLOYS.

Nominal Composition	Endurance Limit, psi.
Plain admiralty	19 000
Admiralty, 0.03 As	17 000
Admiralty, 0.03 Sb	18 000
Admiralty, 0.03 P	17 000

CONCLUSIONS

Laboratory tests indicate that dezincification of alpha brass alloys may be effectively suppressed by a small addition (for example, 0.03 per cent) of arsenic, antimony or phosphorus, without impairment of the alloys in other respects. These conclusions have been confirmed, especially in the case of arsenic, by extensive service experience.

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[8] See p. 874.

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(51) S. L. Hoyt, "Notched-Bar Testing and Impact Testing," Symposium on Impact Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 41 (1938).

(52) D. J. McAdam, Jr., and R. W. Clyne, "The Theory of Impact Testing: Influence of Temperature, Velocity of Deformation, and Form and Size of Specimen on Work of Deformation," *Ibid.*, p. 112.

(53) Sam Tour, "Utility and Non-Standard Impact Testing," *Ibid.*, p. 25.

DISCUSSION

MR. A. W. TRACY[1] (*presented in written form*).—Mr. Lynes has done a thorough job in studying the effect of inhibitors on the dezincification and physical properties of brass and admiralty metal. Our laboratory tests confirm Mr. Lynes' conclusions that arsenic, antimony, and phosphorus are equally effective in preventing dezincification of admiralty.

Our tests were made in one per cent cupric chloride solution by total immersion at room temperature for 1 yr. and at 120 F. for six months. Test specimens were strips of sheet metal 0.050 in. thick annealed at 500 C. Tension tests were made on uncorroded material and on corroded strips at two-month intervals.

Uninhibited admiralty metal was corroded by plug-type dezincification. The inhibited alloys were almost uniformly corroded. The uninhibited admiralty metal rapidly lost tensile strength and ductility, especially in the 120 F. test. The tensile strength and ductility of the three inhibited alloys dropped much less rapidly and there was no significant difference between the admiralty metal with arsenic, antimony, or phosphorus. The physical test results, showing percentage of original tensile strength and percentage of original ductility are given in the accompanying Table I.

It is of interest to record that several million pounds of arsenical admiralty metal condenser tubes have been placed in service since this modification was introduced in 1933. These tubes have shown excellent resistance to corrosion by dezincification and have shown the same degree of resistance to other types of corrosion expected of admiralty tubes.

TABLE I.—TESTS IN 1 PER CENT CUPRIC CHLORIDE.

	Room Temperature—12 months		120 F.—6 months	
	Percentage of Original Tensile Strength	Percentage of Original Ductility	Percentage of Original Tensile Strength	Percentage of Original Ductility
Uninhibited admiralty	19.4	18.5	37.5	17.5
Admiralty + 0.03 per cent arsenic	73.8	80.8	79.1	92.8
Admiralty + 0.07 per cent antimony	72.3	87.6	88.9	97.6
Admiralty + 0.05 per cent phosphorus	64.5	81.4	89.0	100.0

MESSRS. ALAN MORRIS[2] AND R. S. PRATT[2] (*presented in written form*).—We would like to add a little to the work that has been reported on the effect of arsenic in Muntz metal. Comparative tests were made by loss of strength methods after immersion of strip samples in an acid well water containing about 80 ppm. of free carbon dioxide. Tests were made after one year immersion in cold well water and two months in hot well water (160 to 170 F). The results of these tests are as follows:

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[1] Assistant Metallurgist, The American Brass Co., Waterbury, Conn.

[2] Chief Metallurgist, and Metallurgist, respectively, Bridgeport Brass Co., Bridgeport, Conn.

Arsenic, per cent	Tensile Strength, psi.		Loss in Strength, per cent
	Original	Final	
AFTER 1 YR. IN COLD WELL WATER			
0	53 350	39 050	26.7
0.03	55 300	48 750	11.8
0.07	55 500	55 600	none
0.11	58 250	58 550	none
0.21	58 800	58 900	none
AFTER 2 MONTHS IN HOT WELL WATER			
0	53 350	completely dezincified	100
0.03	55 300	43 400	21.5
0.07	55 500	51 800	6.7
0.11	58 250	54 900	5.7
0.21	58 800	56 000	4.8

These tests indicate that arsenic is also beneficial in reducing dezincification in Muntz metal as well as in admiralty metal or 70–30 brass. It is evident, however, that best results are obtained when the quantity of arsenic is greater than that usually added to admiralty metal.

MR. F. M. BARRY[3] (*presented in written form*).—The author has chosen for corrosion tests in the laboratory a 5 per cent cupric chloride, 5 per cent hydrochloric acid solution, the reason being that previous work had demonstrated its suitability for test purposes. This solution is strongly oxidizing in character and of a high degree of acidity, its pH value being less than 1.0. It is essentially a solution which will attack and dissolve copper alloys rapidly and uniformly. In my opinion, this solution is entirely too active as an accelerated corrosion testing agent and bears no relation or similarity to actual service conditions under which dezincification of brass usually occurs.

Under the heading (*b*) Intergranular Corrosion, page 869, the first paragraph deals with the reported intergranular corrosion of arsenical admiralty metal in laboratory tests by Mitchell and by Barry, working independently of each other. Mr. Lynes stated that this type of intergranular penetration must not be confused with the well-known season cracking of brass which is intergranular in character but requires simultaneous action of stress and corrosion. Season cracking is generally defined as a crack due to the simultaneous action of internal tensile stress and certain specific corrosive media, for example, ammonia or ammonium compounds or mercury and mercury compounds. Mercury and ammonium compounds are the only ones known to bring about this type of cracking of brass. The corrosion tests of Mitchell and Barry indicate that, in the case of alpha brasses containing arsenic, corroding agents other than mercury and ammonium compounds will attack the metal intergranularly even in the absence of stresses in the metal, and cracking of such brasses even under mild stressing would be expected which might be identified in many cases as season cracking or stress corrosion cracking.

On page 870, the author states that Mitchell and Barry infer that arsenic is a potentially dangerous inhibitor, responsible for service failures. In regard to the Barry reference, and noting that Mr. Lynes has used the word "infer" to mean "conclude" he has misinterpreted the subject matter of my paper in some respect. Quoting the paper in question the most damaging statement that can be found regarding arsenical admiralty metal is the following: "The susceptibility of arsenical admiralty metal to intergranular corrosion may explain a number of service failures of arsenical admiralty metal tubes, as a result of cracking, which have come to our attention." In other words, the Barry paper suggested only the possibility of some relation between cracking of admiralty metal tubes and the arsenic content of the metal. No conclusions were made contrary to Mr. Lynes' statement.

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[3] Chemist, Scovill Manufacturing Co., Waterbury, Conn.

Mr. Lynes believes that over emphasis has been placed by the above-mentioned investigators (Mitchell and Barry) on cracking of admiralty tubes. As a matter of fact, the paper by Barry was devoted almost entirely to a description of a test procedure for evaluating the dezincification resistance of brass alloys followed by a discussion of results of tests on a number of copper alloys, and the reference to the intergranular corrosion of arsenical admiralty metal was incidental to the discussion of test results on all alloys under test. In view of the statements on page 870, in regard to laboratory corrosion tests and "the danger of indiscriminate use of short-time tests and the careless or misleading application of the test data" which are made in connection with a discussion of Mitchell and Barry's work, I wish to quote the summary given in Barry's paper and ask Mr. Lynes to point out any "indiscriminate use of short-time tests or the careless or misleading application of the test data" contained in this summary:

> "1. A corrosion test for measuring the dezincification resistance of copper-zinc alloys has been described, and results of such tests on a number of alloys have been given.
>
> "2. The tests indicate that the addition of phosphorus, antimony or arsenic to admiralty metal in amounts up to approximately 0.05 to 0.06 per cent will inhibit dezincification under the particular conditions of the test.
>
> "3. Intercrystalline corrosion of arsenical admiralty metal has been revealed by the cupric chloride test, particularly at elevated temperatures (120 F.).
>
> "4. The cupric-chloride test is useful only in determining the dezincification resistance of brasses. The results of this test cannot be used to predict the behavior in service of such alloys when subjected to other forms of corrosion."

If Mr. Lynes has any objections to the above summary of the Barry paper, or if he has any constructive criticism to offer in regard to the paper as a whole, the author would be pleased to receive it. The type of criticism offered in the present paper by Mr. Lynes, however, is neither constructive nor helpful and contributes nothing to the subject at hand.

In speaking of the conclusion, attributed to Mitchell and Barry, that arsenic is a potentially dangerous inhibitor, responsible for service failures, the author states that it appears to be unsubstantiated either in the literature or by practical experience. In this connection, my paper referred to an article by E. S. Dixon on "Service and Life of Non-Ferrous Tubes in Petroleum Refining," appearing in the ASTM BULLETIN.[4] The following remarks regarding nonferrous alloys (brasses) containing arsenic are quoted from this paper:

"The author has noted an improvement with respect to dezincification when arsenic was added, but because of cracking of tubes containing arsenic the use of this alloy tube has been discontinued. It is impossible to state whether the cracking of tubes occurred because of the arsenic. Many tubes did not crack although arsenic was present." Evidently, Mr. Lynes has completely discounted these observations by E. S. Dixon.

Analysis of the cause of failures of 46 admiralty metal heat exchanger tubes from diverse sources, examined by Mr. Lynes' laboratory and reported on page 870 of the paper showed that 22 per cent of the tubes had failed as a result of cracking. Arsenic in commercial amount was contained in 23 per cent of the tubes examined. Mr. Lynes has identified 15 per cent of the cracks as due to corrosion fatigue, 7 per cent to stress-corrosion and none to intergranular corrosion. I should like to ask Mr. Lynes what percentage of the cracked tubes contained arsenic in view of the fact that the percentage of arsenical tubes is so close to that of the percentage of cracked tubes. Mr. Lynes states that cracking is not a major cause of tube failure. From the 46 tube failures investigated by Mr. Lynes, however, 23 per cent of such failures as a result of cracking seems to be a substantial proportion of the total failures and would undoubtedly be considered very serious from the standpoint of the user of condenser tubes. In his analysis of tube failures as a result of cracking, I should like to ask the author how he distinguishes between tube failures due to stress corrosion and those due to intergranular corrosion since both would show an intergranular type of fracture. I believe that Mr. Lynes will agree that, if intergranular corrosion of arsenical brasses does occur under certain conditions of service, actual failure of such brasses would not be likely to occur simply through continued corrosion of this type until it extended through the tube wall because tubes are usually subjected to stresses in service which would come into play simultaneously with the intergranular corrosion and, depending upon the type and magnitude of such stresses, actual failures might well be diagnosed as due to stress-corrosion or to corrosion-fatigue. There seems to be some question in Mr. Lynes' mind on this point and it is one of his chief arguments for refusing to consider that there may be correlation between the behavior of arsenical admiralty metal in service and those laboratory tests which have shown the susceptibility of arsenical admiralty metal to intergranular corrosion.

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[4] ASTM BULLETIN, No. 102, January, 1940, p. 21.

The author has referred several times throughout the paper to published reports of Mitchell and of Barry on the intergranular corrosion of arsenical admiralty metal and has attempted to show by tests of his own that intergranular corrosion is relatively unimportant or does not exist. In cases of this kind where certain test results are questioned, the natural procedure would be, in our opinion, to check the work of the earlier investigators, adhering as closely as possible to their methods of test, to find out whether their test results can be duplicated. Has Mr. Lynes attempted to follow through either the laboratory corrosion test of Mitchell or of Barry and, if so, has he been able to check their reported results? Also, it is pertinent to inquire for what reasons Mr. Lynes prefers to place more reliance on his own accelerated tests than those of Mitchell and of Barry.

I am as interested as Mr. Lynes to see more light shed on this controversial subject of the intercrystalline corrosion of arsenical admiralty metal. In spite of the large number of tests conducted and reported by the author of this paper, however, the subject in my opinion still remains open for considerable future investigational study before any definite or positive conclusions can be reached regarding it.

MR. W. LYNES[5] (*author's closure*).—It is interesting to note that tests and observations made by Mr. Tracy are confirmed in the present paper.

Messrs. Morris and Pratt have added to the meager available information on the effect of arsenic in Muntz metal by presenting results of tests in an acid well water containing about 80 ppm. of free carbon dioxide.

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[5] Research Metallurgist, Research Dept., Revere Copper and Brass, Inc., Rome, N. Y.

Mr. Barry believes that the test solution of 5 per cent cupric chloride, 5 per cent hydrochloric acid used by the author is entirely too active, and bears no relation to actual service conditions. The test purposely was made drastically accelerative, so that freedom from dezincification under the test conditions would be strongly indicative of similar freedom under any operating condition. On the other hand, had some particular set of operating conditions been simulated, the dezincification resistance of the alloy under more severe conditions could not be inferred. The well-known mercurous nitrate test for season cracking is based on analogous considerations.

In regard to the statement in the present paper that "Mitchell and Barry infer that arsenic is a potentially dangerous inhibitor, responsible for service failures," the author is glad to have Mr. Barry's correction that his paper "suggested only the possibility of some relationship between cracking of admiralty metal tubes and the arsenic content of the metal." However, Mitchell unequivocally has stated that intercrystalline corrosion of arsenical admiralty tubes has occurred in service.[6] In this connection it is interesting to note that Mr. Barry, in his discussion, has conceded the impossibility of demonstrating conclusively that intergranular corrosion ever occurs under service conditions, inasmuch as some stress is always present.

Mr. Barry, in support of his supposition that intergranular corrosion may explain a number of service failures of arsenical admiralty metal tubes, has quoted from an article by Dixon.[4] The following remarks are quoted: "The author has noted an improvement with respect to dezincification when arsenic was added, but because of cracking of tubes containing arsenic the use of this alloy has been discontinued. It is impossible to state whether the cracking of tubes occurred because of the arsenic. Many tubes did not crack although arsenic was present." The foregoing merely states that arsenical admiralty tubes may crack in service, which is entirely expected, as it has never been suggested that addition of arsenic to admiralty metal precluded such a possibility. Furthermore, there is no necessary relationship between cracking of condenser tubes and intergranular corrosion. In the experience of the author, the most common cause of tube cracking is corrosion-fatigue. This type of failure cannot well be confused with intergranular corrosion, because the path of fracture in corrosion-fatigue is transgranular. The author believes that, on a probability basis, the cases of cracking cited by Dixon are most likely to have been the result of corrosion-fatigue, and to bear no relation to the presence or absence of arsenic.

In reference to an analysis of the cause of failure of 46 admiralty metal heat-exchanger tubes examined at the laboratory with which the author is associated, in a 3-yr. period, Mr. Barry asks what percentage of the cracked tubes contained arsenic. The answer is 40 per cent. He also asks how tube failures due to stress-corrosion are distinguished from those due to intergranular corrosion. Two plain admiralty tubes and one arsenical admiralty tube out of 46 admiralty tubes examined showed an intergranular type of failure. As regards the plain admiralty tube-failures, one tube "season-cracked" where rolled into the tube sheet, the other at a flattened U-bend; that is, in both cases, the stresses necessary for stress-corrosion cracking were developed during assembly of the heat-exchanger. The arsenical admiralty tube which failed by stress-corrosion cracking had been accidentally in contact with mercury, as shown by the examination. As is well known, mercury is particularly active in producing cracking of stressed brass tubing. In this case, the cause of the stress factor is not known. Thus in the 46 tubes examined, none showed evidence of having failed from intergranular corrosion. To answer the above question in a general way, stressless intergranular corrosion would evidence itself by widespread intergranular corrosion. In the case of 18-8 stainless steel this is known as "sugaring," and tends to reduce the test specimen to its component grains. In the case of stress-corrosion cracking where the stress intensity was high, the intergranular corrosion would be localized at a crack or at several cracks. At lower stress intensities, the end result would, in appearance, tend to approach that of intergranular corrosion.

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[6] N. W. Mitchell, "Recent Developments in Copper Alloy Condenser Tubes," *Oil and Gas Journal*, July 27, 1939, pp. 87–88.

Mr. Barry states that Mr. Lynes "has attempted to show by tests of his own that intergranular corrosion is relatively unimportant or does not exist." This is somewhat of a misinterpretation of the author's position. A more careful reading of the paper would show that the validity of the results obtained by Mr. Barry in laboratory tests have not been questioned, nor have tests made by the author been offered as evidence that intergranular corrosion of arsenical admiralty metal cannot occur under appropriate conditions. The essence of the author's position is that extended service experience with large tonnages of arsenical admiralty metal tubing has shown no propensity toward intergranular corrosion of these tubes in service, and that but a small percentage of these tubes have failed intergranularly, irrespective of the type of intergranular failure. The author still maintains that valid evidence is lacking to show that the intergranular corrosion obtained by Barry and Mitchell in laboratory tests has its counterpart under operating conditions. At the moment, the test results of Barry and Mitchell are scientifically interesting but lack proven commercial significance.

The author would like to thank the various parties who, by their discussions, have added to the value of the paper.

THE PROPERTIES OF CERTAIN LEAD-BEARING ALLOYS

BY ALBERT J. PHILLIPS,[1] ALBERT A. SMITH, JR.,[1] AND PAUL A. BECK[1]

SYNOPSIS

A new lead-base bearing alloy containing approximately 12.5 per cent antimony, 3 per cent arsenic, 0.75 per cent tin, has been developed and its properties have been compared to those of conventional lead-base and tin-base alloys. At elevated temperatures, approximating those prevailing in automotive engine bearings, the tensile strength and Brinell hardness of the new alloy are considerably higher than those of the standard lead-base alloy and equal to those of the tin-base alloy. In addition, it retains its higher hardness over long periods of exposure to elevated temperatures, whereas the standard alloys show considerable softening under such conditions. The rotating-beam fatigue strength and the flexure fatigue strength of the new alloy at room temperature are superior to those of the standard bearing alloys. This superiority is also fully retained at 100 C. The alloy is easy to cast, retains its composition after repeated remelting, and shows relatively little loss of weight due to drossing. Above 175 C. the alloy is ductile enough to permit shaping as required in certain bearing manufacturing processes. The solidus temperature is 242.5 C. and liquidus temperature 295 C. The properties of the new alloy indicate that it is superior to the lead-base and tin-base bearing alloys now in general use. Since tin is a strategic material, the new alloy promises to be of great interest because its use can decrease the amount of tin employed for bearing purposes.

Some time ago in the Central Research Laboratory of the American Smelting and Refining Co. a program was started for the expressed purpose of determining or developing the best lead-base bearing alloy available. From a preliminary survey of the data given in the literature it would seem, on the face of it, that lead-base bearing alloys are quite the equal and, in some cases, possibly superior to tin-base bearing alloys but as a matter of practical experience this has not proved to be the case.

Tin-base bearing alloys have usually proved their superiority, particularly in automotive engines where the bearings are subjected to elevated temperatures. Since the data in the literature cover very largely the properties of lead-base bearing alloys at room temperatures it was anticipated that the failure of the physical properties to indicate the relative merit of tin- and lead-base bearing alloys was due to the measurement of these properties only at room temperatures. Therefore the properties of the various accepted bearing alloys were measured over a range of temperatures wide enough to include the highest temperature at which bearings could normally be employed. This procedure did indeed indicate that the properties of most lead-base bearing alloys fall off more rapidly with increasing temperature than do the properties of tin-base bearing alloys. A further factor was soon discovered which proved to be of greater significance and that is—time at the elevated temperature. When bearing alloys were held, for example, at 150 C. for a period of many days it was found that with all the commercial lead-base alloys tested the properties fell off much more rapidly with time than did the properties of the conventional tin-base alloys. As a result of this survey it was felt that if a lead-base alloy could be developed that would retain its physical properties over a range of temperature and time at least equal to the best tin-base alloy, the application of lead-base alloys could be widened to a great extent and very largely displace tin-base bearing alloys. Since tin is a high-priced strategic metal, this possibility is of considerable significance at the present time.

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[1] Research Metallurgists, American Smelting and Refining Co., Barber, N. J.

SURVEY

As not one of the lead-base bearing alloys tested had properties reasonably close to those of the tin-base bearing alloys which were set as the minimum target for the development, a survey was made of all lead alloy systems that could possibly be of use for bearings. The initial survey was simple, since it merely took into account the binary systems of lead with all other elements of the periodic table. The systems of many elements are not known and for the most part such systems can be eliminated since they contain very largely either the rare elements which are not available at a reasonable price or elements that are difficult, if not impossible, to alloy with lead. Of this group only two or three systems were investigated, none of which proved to be promising.

Of the group where the constitution is known, most of the systems can be eliminated without performing any experimental work. For example, the eutectics of platinum, gold and other precious metals can be eliminated on the basis of cost. Other systems having low melting eutectics can be eliminated on the basis that their melting point is too close to the working temperature desired. Certain other elements like lithium, sodium, potassium, etc., are much too reactive to permit the formation of an alloy stable enough for regular commercial usage. Hence, of the simple eutectic systems only two or three were investigated and in no case was an alloy found that had satisfactory properties.

Of the group of alloys that showed a decreasing solid solubility with temperature, which of course are potentially hardenable alloys, fairly careful surveys have been made in the past and consequently relatively little work was done on these alloy systems considering the number of potentially interesting systems. One factor that eliminated a great many of these alloys was the instability at temperatures approaching 150 C. of the hardness due to precipitation. In other words, while these alloys do produce desirable properties at room temperature and even at elevated temperatures for a short period of time, when exposed to elevated temperatures for long periods of time their properties approach those of common lead.

It was therefore felt after performing only a moderate amount of work on the binary systems that the possibility of developing a satisfactory binary lead-base alloy for bearings was quite remote and that there was more chance of success in developing an alloy which depended for its strength upon a matrix material consisting of a ternary or polynary eutectic. While the number of possibilities in ternary and polynary systems is tremendous, yet there is one fact that makes a survey of such systems for bearing alloys rather simple and that is—the alloy developed must have a reasonably high melting point if good physical properties are to be obtained at elevated temperatures. At the same time the alloy must contain a relatively large amount of the alloying elements if a substantial effect on the properties is to be obtained. However, even with these limitations, the number of alloys that were to be investigated was so great as to preclude making a complete survey of the properties of these alloys. For the purpose of quickly testing the series it was decided that a simple test, that could be conducted with a minimum of outlay for test specimens and testing procedure, would be employed and that all systems which passed this preliminary survey would be further investigated. The test procedure employed in this preliminary survey was to determine the Brinell hardness over a range of temperatures and a reasonable period of time.

After a great many preliminary tests, a lead-base alloy system was developed which proved to be at least equal to the present commercial tin-base bearing alloy in practically all of its physical properties. It is the purpose of this article to describe the properties of this alloy, comparing them with those of the conventional lead-base and tin-base alloys. The alloy[2] as finally developed had the following composition:

Antimony, per cent	12.5
Arsenic, per cent	3
Tin, per cent	0.75
Lead, per cent	balance
Solidus point	242.5 C.
Liquidus point	295.0 C.

It was found that other elements could be added to this alloy system without deterioration and in some cases with minor improvements in properties. These elements were, for example, copper, bismuth, cadmium, nickel, mercury, etc. In no case was it found that small amounts of the various addition agents tested developed any serious loss in desirable properties. Consequently it is felt that the alloy was quite insensitive to impurities or contaminating elements, a feature that is of some commercial importance.

The effect of additions of arsenic to lead-base bearing alloys has been studied by a number of investigators[3] and the results show improved high-temperature properties and considerable grain refinement. In addition, Schwarz[4] states that the addition of arsenic to lead-base bearing alloys markedly lowers the coefficient of friction. However, the above quaternary system has not previously been completely surveyed to determine the composition ranges that give the best combination of properties.

BRINELL HARDNESS

Test specimens were prepared for Brinell hardness tests by alloying the various constituents in Dixon "graphite" crucibles, heating to 400 C. for the lead alloys and 500 C. for the tin-base alloys, and casting in an open iron mold to produce slabs 7 in. long, 1⅛ in. wide and ¼ in. thick. The mold was used cold and was quite heavy in order to produce the maximum degree of chill, a condition that prevails in the commercial casting of bearings. All hardness impressions were made on the smooth bottom surface of the castings.

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[2] U. S. Patent No. 2,232,185, February 18, 1941.

[3] Metals Handbook, 1939 Edition, p. 1554.
H. J. Roast and C. F. Pascoe, "How Arsenic Affects Alloys," *The Foundry*, Vol. 51, pp. 67–70 (1923).

[4] M. von Schwarz, "Tests on Frictional Properties of New Bearing Alloys, Particularly Lead-Base Alloys," *Zeitschrift für Metallkunde*, Vol. 28, p. 128 (1936).

Brinell hardness impressions were made as soon after casting as was practical, and after several readings had been obtained to give an average value, the castings were placed in an electric oven automatically controlled at 150 C. At the end of various periods of time, the samples were removed from the oven to an oil bath automatically controlled at 150 C. and which was mounted on the anvil of a Brinell machine. After taking two or more impressions of each specimen at 150 C. the specimens were removed from the oil bath and allowed to cool to room temperature in order to obtain the room temperature Brinell hardness in the annealed condition. After the room temperature impressions had been made and the values of the impressions both at room temperature and 150 C. were recorded, the specimens were returned to the electric oven for further heat treatment.

TABLE I.—BRINELL HARDNESS AS RELATED TO TEMPERATURE.

Composition, per cent					Brinell Hardness					
						At 30 C.		At 150 C.		
Antimony	Arsenic	Tin	Copper	Lead	As Cast	After 7 days at 150 C.	After 46 days at 150 C.	After 17 hr.	After 7 days	After 46 days
12.65	2.97	0.65	....	Bal.	22	20.5	19.5	10	9.7	9.3
15	0.2	5	0.5	Bal.	20	16.5	15.5	7.3	7	5.8
13.89	0.34	9.42	0.44	Bal.	22.3	16.5	....	7	6.6	...
6.25	....	88.25	5.5	....	25	22.3	20.9	9.5	9.3	8.3

The Brinell hardness tester employed a 10-mm. ball and was a direct-acting dead-weight machine employing either 25, 50, 75, or 100-kg. loads. The load was applied for a period of 30 sec. and it was found necessary to control this period rather precisely. As a general rule the scatter in readings obtained on any one specimen seldom exceeded one Brinell number. Values obtained with the above procedure are listed in Table I.

TENSILE PROPERTIES

Tensile properties were determined on the various bearing alloys in the "as cast" condition. The alloys were prepared in the same way as for the Brinell specimens but were chill cast into a cold iron mold. The iron mold was shaped to produce flat specimens with a reduced gage length 2 in. long by 1 in. wide by $\frac{1}{8}$ in. thick.

The tension tests at elevated temperature were conducted using a universal hydraulic testing machine of the lapped ram type equipped with two Bourdon gages, one reading to 300 lb. and the other 1000 lb. The tests were conducted in an electrically heated oil bath automatically controlled to within one or two degrees of the designated temperature. After a specimen had been clamped in the grips and was placed in the bath, a period of 20 min. was allowed for it to reach temperature before testing was commenced. The speed of the head was 0.05 in. per inch of gage length per minute. All values as reported are the results of at least two

TABLE II.—TENSILE PROPERTIES AT VARIOUS TEMPERATURES.

Composition, per cent					25 C.		100 C.		150 C.		200 C.	
Antimony	Arsenic	Tin	Copper	Lead	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent
12.6	3	0.75	...	Bal.	9 800	1.5	6700	4	4200	10	1900	70
15	0.2	5	0.5	Bal.	10 000	5	5400	27	2900	55	1200	100
6.25	...	88.25	5.5	....	10 600	6	5900	27	3400	33	1700	50

closely checking tests. Data from specimens containing defects in the fracture were discarded in all cases. The data are given in Table II.

DROSSING TESTS

The commercial use of any bearing alloy can be very considerably influenced by the composition stability of the alloy under normal conditions of commercial operation. For example, an alloy that contains elements which readily volatilize or oxidize to form dross and thus change the composition significantly are looked upon with disfavor on the part of operating men due to the nuisance of continually checking compositions. Such difficulties may be so great as to increase the cost prohibitively due to rejected material from off-composition. Consequently a series of comparative drossing tests was conducted in order to determine whether the new alloy was as stable in composition as the conventional tin- and lead-base bearing alloys. 500 g. of each alloy was melted in a porcelain casserole and held at 400 C. for 30 min. During this period a stirrer, skimming the surface of the metal in a circle $1\frac{1}{2}$ in. in diameter, was rotated at exactly 100 rpm. Samples taken before and after this drossing period were analyzed with the results given in Table III.

TABLE III.—CHANGE IN COMPOSITION DUE TO DROSSING.

	Lead Babbitt		Tin Babbitt		New Alloy	
	Initial	Final	Initial	Final	Initial	Final
Antimony, per cent	13.9	14.0	4.75	4.65	13.38	13.18
Arsenic, per cent	0.24	0.24	....	....	3.22	3.28
Copper, per cent	....	....	5.43	5.43	....	....
Tin, per cent	6.62	6.67	Balance	Balance	0.66	0.74
Lead	Balance	Balance	....	....	Balance	Balance
Total weight, g.	500	360	500	174	500	406

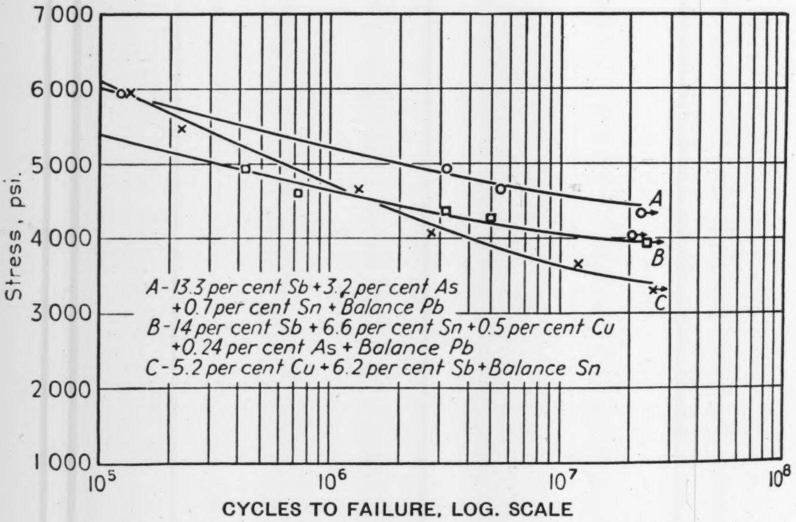

FIG. 1.—Fatigue Test Results on the Various Bearing Alloys.

In addition a great many tests were conducted under operating conditions when actually casting bearings and in no case was it necessary to adjust composition even after several days of casting and remelting scrap specimens.

FATIGUE TESTS

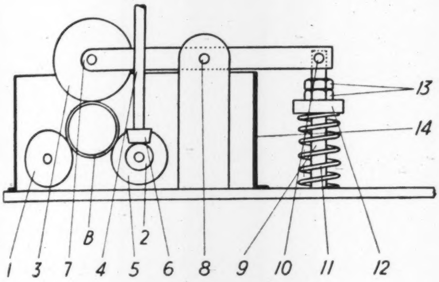

FIG. 2.—Fatigue Test Machine.

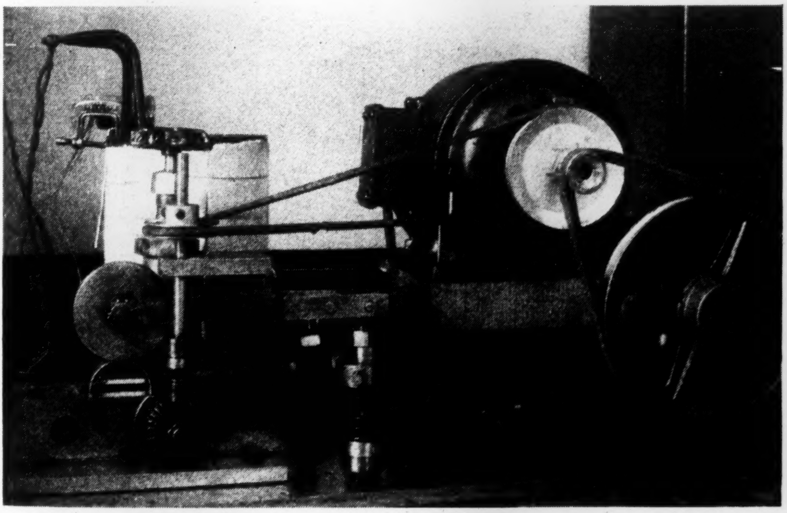

FIG. 3.—Fatigue Test Machine.

Fatigue strengths of the various alloys were determined by conventional means. Slugs $\frac{3}{4}$ in. in diameter by 6 in. long were cast in a cold iron mold. These castings were machined into the 0.350-in. diameter standard test specimens for R. R. Moore rotating-beam fatigue machines. The machines were specially balanced in order to permit testing with low loads but otherwise were standard design. They were run at 2000 rpm. and the average temperature obtained by the specimens was about 37 C. The results obtained with the various bearing alloys are given in Fig. 1. It will be noted that on the basis of 20 million cycles the fatigue strength of the conventional tin babbitt is 3400 psi., of the conventional lead babbitt 3900 psi., and of the new lead alloy 4400 psi. While a high fatigue strength at room temperature probably does indicate to a reasonable extent a desirable property in a bearing alloy, the fatigue value at the operating temperature of the bearing is of course much more important and may be of quite different magnitude. Likewise in automobile bearings the fatigue strength of the bearing alloy is only of importance if the fatigue strength of the bond fastening the bearing alloy to the steel backing is satisfactorily high. Consequently a special testing machine to determine these two properties was developed on the basis of a description of a testing machine used by a European manufacturer of automobile engines.[5] The machine consists of a device for rotating and flexing a bushing made by centrifugally casting the bearing alloy under test into a steel ring of proper dimensions. The ring is mounted on two rollers which rotate at the desired speed. A third roller presses the top of the ring, flexing it the desired amount. The machine is illustrated diagrammatically in Fig. 2 in which *B* is the test specimen, rolls 1 and 5 are the bottom rolls for supporting the specimen, 6 and 2 are the gears for driving one of these rolls, 3 is the roll for applying the load, which is supplied by spring 11 acting on the lever arm 7. The apparatus as built is illustrated in Fig. 3. In practice the machine is flooded with oil which is circulated through a suitable heating tank where it is maintained automatically at the desired temperature. The specimens were driven at 1190 rpm. and there were, quite obviously, three flexing cycles per revolution. The test procedure was to examine each specimen periodically by removing it from the machine to determine whether failure had taken place. This could be detected in two ways. First, by ringing the specimen and, second, by visual examination for cracks. Usually the ring test would detect cracks before they could be seen with the unaided eye. The change from a clear bell-like tone to a "cracked bell" sound was quite marked. The bearing bushings were prepared by centrifugally casting the various alloys into cold-drawn seamless steel tubing. The tubes were tinned by placing them in a bath of the tinning alloy covered with a layer of molten zinc chloride and maintained at about 450 C. This high temperature was necessary to prevent the relatively heavy steel tubing from chilling down the molten tinning alloy to the freezing point. The tubes for both lead alloys were tinned with an alloy containing 12 per cent antimony, 4 per cent tin, and 84 per cent lead. The tinning bath for the tin babbitt alloy was of the same composition as the alloy. All test specimens were machined to 2 in. outside diameter, 1 in. wide, 0.040 in. steel thickness and 0.020 in. bearing metal thickness. Table IV gives the data obtained at both room temperature and 100 C.

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[5] Private communication.

DISCUSSION

As has been mentioned before, the Brinell hardness data, particularly after a period of 7 to 46 days at 150 C., reveal that the physical properties, at least as determined by hardness, of the conventional lead-base bearing alloys are decidedly inferior to those of the standard tin-base alloy. However, the new lead-base bearing alloy retains its hardness to a greater extent than does the tin babbitt. The tensile strength data confirm the hardness data and show that while the new alloy is initially a trifle softer at room temperature than the lead or the tin-base babbitt, it is very much better than these two alloys at 100 C. and higher. It is true that the ductility of the alloy at room temperature, as determined by elongation, is inferior to that of either the lead- or tin-base babbitts but for most bearing

TABLE IV.—FLEXURE TESTS AT VARIOUS LOADS AND TEMPERATURES.

Composition, per cent					Temperature, deg. Cent.	Load, lb.	Cycles to Failure
Antimony	Arsenic	Tin	Copper	Lead			
6.2	....	Bal.	5.5	....	30	38	210 000
6.2	....	Bal.	5.5	....	30	24	860 000
10	0.25	5	...	Bal.	30	24	1 070 000
13	3	0.75	...	Bal.	30	38	960 000
13	3	0.75	...	Bal.	30	24	2 600 000
6.2	....	Bal.	5.5	....	100	20	110 000
6.2	....	Bal.	5.5	....	100	15	430 000
10	0.25	5	...	Bal.	100	20	210 000
10	0.25	5	...	Bal.	100	15	1 500 000
13	3	0.75	...	Bal.	100	20	430 000
13	3	0.75	...	Bal.	100	15	3 200 000

purposes ductility is an unimportant factor and is useful only in that it is supposed to be a guide to fatigue or flexure strength. It is important for making bearings by certain procedures that the alloy have a reasonably wide "working temperature" range to permit shaping without fracture. However, in the temperature range of 175 to 200 C. the new lead-base bearing alloy is ductile enough to permit the most drastic shaping operation.

The composition stability of the new alloy is most satisfactory and in addition the low rate of dross formation promotes economical utilization of the alloy. In fact the alloy is far easier to handle in the molten state than any bearing alloy tested. The high fatigue strength as shown by the tests on the R. R. Moore rotating-beam machine was encouraging but not conclusive evidence that the alloy would show real superiority in this respect in commercial bearings. However, the flexure test machine demonstrated such a marked improvement over the conventional lead-base or tin-base babbitts that there is little question that bearings made of the new alloy have improved fatigue resistance. In addition there have been several installations of the alloy in automobiles and machinery and no failures have been experienced to date. Several of these tests have now run over 18 months and it is felt that they afford reasonable confirmation of the results predicted from the laboratory tests.

DISCUSSION

MR. H. W. GILLETT[1] (*presented in written form*).—It is encouraging to see that an attempt is being made to reproduce, in a lead-base babbitt, the properties of tin-base babbitt, both at room temperature and at operating temperatures. Many experimenters have "improved" lead-base babbitt by hardening it at room temperature, probably beyond the degree dictated by embedability requirements, without improving its properties at operating temperature. Until someone can more clearly define what properties are needed in babbitt, there is always the possibility that what the shaft wants is just what tin-base babbitt gives, neither more nor less.

Working on the assumption that a similar hardness-temperature curve (determined on aged material) to that of tin-base babbitt would indicate a first approach to the required properties and that good bonding, or soldering, power was essential, we have been studying substitute lead-base babbitts at Battelle. Since silver confers strength at elevated temperature, since lead-silver alloys are recognized solders, and since the United States has large amounts of silver not being put to any useful engineering purpose, silver was not excluded from the list of alloying elements tried.

From this work it was found that a lead-base alloy with $2\frac{1}{2}$ to 5 per cent silver, 15 per cent antimony, 2 per cent tin, 0.25 per cent copper has many similarities to tin-base babbitt. One could consider this a cross between No. 10 lead-antimony-tin babbitt of the Standard Specifications for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 - 26),[2] which has bearing properties but low strength at high temperature, and a lead-silver-copper solder which has high-temperature strength. The tin content was put in primarily because some tin in a lead-base bearing is considered helpful against corrosion by oil, and while it is probably not needed for that purpose in the 5-component alloy, it was retained and held at this level because of its strengthening effect and because alloys of either much higher or much lower tin tend to soften more on aging.

We used soft and hard tin-base babbitts of $7\frac{1}{2}$ per cent antimony, $3\frac{1}{2}$ per cent copper, and $8\frac{1}{3}$ per cent antimony, $8\frac{1}{3}$ per cent copper for comparison. These and the lead-base babbitts studied were tested for hardness as follows: at room temperature as cast; at room temperature after aging 24 hr. at 300 F. using a $2\frac{1}{2}$-mm. ball, loaded for 30 sec., 5-kg. load; and at 300 F. after aging 24 hr. at 300 F., with $2\frac{1}{2}$-mm. ball, 2-kg. load, for both 30 sec. and 300 sec.

Composition, per cent					Room Temperature, 30 sec.		Aged 24 hr. at 300 F. and Tested at 300 F.	
Tin	Lead	Antimony	Copper	Silver	As cast	After 24 hr. at 300 F.	30 sec.	300 sec.
Balance	....	$8\frac{1}{3}$	$8\frac{1}{3}$	....	20	20	7.2	5.5
Balance	....	$7\frac{1}{2}$	$3\frac{1}{2}$	....	19	17	6.0	4.4
2	Balance	15	$\frac{1}{4}$	5	23	21	8.9	6.6
3	Balance	10	—	2.6	21	18	7.2	5.2

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[1] Metallurgist, Battelle Memorial Inst., Columbus Ohio.

[2] 1939 Book of A.S.T.M. Standards, Part I, p. 677.

Our 300 F. hardness values for the tin-base babbitts are lower than those of Phillips, Smith and Beck, for an intermediate composition, which probably is due to differences in casting conditions. We did not follow up arsenic-containing alloys very far because of indications of inferior bonding power compared to the silver-containing alloys.

Cylinders of the lead-base babbitts containing silver could be hammered down 40 per cent or more before cracking started, so formability seems adequate.

The raw material cost for these babbitts that contain silver is higher than for those of Phillips, Smith and Beck, but, figuring on industrial silver and present cost of tin, the 5 per cent silver alloy costs two-thirds as much as tin-base, the 2½ per cent silver alloy one-half as much.

We have run Amsler seizure tests on the lead-base alloys with silver and found that their frictional properties are almost identical with those of the tin-base babbitts. Full-size bearing tests, on a machine of the General Motors design for testing connecting rod bearings, have shown that such an alloy resists cracking as well as the tin-base babbitts.

We have run no fatigue tests on such alloys in the rotating-beam machines. In the past, however, we ran a large number of rotating-beam tests on various standard tin-base and lead-base babbitts, both at room temperature and at elevated temperatures. At that time, we found the thing pointed out by Phillips, Smith and Beck, that tests at room temperature were no indication of what the results would be at elevated temperatures, nor did high-temperature fatigue tests prove very illuminating. For that reason, we have gone to the General Motors type of bearing test for obtaining data on propensity toward cracking. We have questioned the value of the flexure test, for the reason that it appears to give data that are not translatable into service. For example, the data in the paper would seem to show that lead-base babbitts, even without arsenic, are more resistant to fatigue-cracking than tin-base babbitts, which seems contrary to general experience.

The every-day lead-base babbitts, in the extremely thin layer now used in automotive bearings, seem to be serviceable as substitutes for the thicker layer of tin-base babbitt of earlier days, but this does not help the man who has to reline a failed thick tin-base babbitt bearing if he cannot get tin in a shortage.

It was with this in view that we attempted to produce a lead-base babbitt with the approximate properties of the tin-base, and with approximately the same bonding power.

It is to be hoped that widespread practical trials will be made of the lead-base alloys with arsenic, of those with silver, and of any others that show promise of being adequate substitutes for tin-base babbitt, because of the strategic importance of such substitution. The strategic aspect makes the work of Phillips, Smith and Beck doubly important and particularly welcome.

MR. JOHN M. LESSELLS.[3]—Tin-base babbitts have been replaced by other types of materials for severe applications such as aeroengine crankshaft and connecting rods. This replacement of tin-base babbitts is also taking place in the automotive field. In view of this, what are the chief application and service condition which the authors have in mind for these babbitts?

Secondly, it has been shown that the fatigue life of babbitts increases as the thickness of the babbitt is decreased to a certain minimum and the question arises whether or not the thickness of the babbitts was varied in the authors' tests.

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[3] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

MESSRS. ALBERT J. PHILLIPS,[4] ALBERT A. SMITH, JR.,[4] AND PAUL A. BECK[4] (*authors' closure, by letter*).—In answer to Mr. Lessells' question regarding the application of the new alloy, there are any number of possible places where a low-cost alloy of excellent properties can be used such as automobiles and rolling mills. Because of the strategic importance of tin at the present time a saving in its use in conventional babbitts is of importance.

The flexure fatigue tests were all made on a babbitt thickness of 0.020 in. and this was not varied for the different alloys, as comparative data were desired. It is well known that the thickness of the babbitt lining has a large effect on fatigue life, but this was felt to be outside the scope of the present work.

Mr. Gillett has presented some interesting data on lead-base babbitts containing silver which show very promising properties. He agrees with the authors that high-temperature properties and their retention after prolonged heating are important for bearing alloys. It is difficult to determine by laboratory tests, such as flexure and hardness, whether a particular bearing alloy is superior to another in actual use. However, such tests do serve as guides and actual service installations have confirmed the laboratory tests to a large extent.

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[4] Research Metallurgists, American Smelting and Refining Co., Barber, N. J.

MERCURY CRACKING TEST, PROCEDURE AND CONTROL[*]

By H. Rosenthal[1] and A. L. Jamieson[1]

Synopsis

This paper presents data accumulated during an investigation made on small arms ammunition cartridge cases and small arms ammunition cartridge case draw pieces. The purpose of this study was to determine the effect of different variables which exist in the mercurous nitrate test for detection of excessive stresses in copper-base alloys.

The variables studied include the preparation of specimens prior to testing, mercurous nitrate and nitric acid concentrations, temperature of solutions, time of test period, and re-use of solutions.

Tests were made on two lots of metal, fabricated at Frankford Arsenal, which had stresses of two different magnitudes. These specimens were subjected to tests in which all variables were held constant except those being investigated. The results of these tests show that test conditions should be controlled as to temperature and composition of the solution, period of immersion, and sample preparation and examination.

The use of mercury salt solutions to detect excessive stresses in copper-base alloys was originated and first applied to inspection of ordnance materials by F. Rogers in 1905.[2] Since that time, the use of mercury salt solutions for detecting stresses has steadily grown until it is now universal.

Most of the literature dealing with this subject is not directly applicable to ordnance inspection. In testing small arms ammunition cartridge cases, for example, a definite number of cases representing a production lot are prepared by pickling in a solution containing 40 per cent by volume of nitric acid (sp. gr. 1.42). The pickled cases are then immediately immersed in a 1 per cent mercurous nitrate - 1 per cent nitric acid aqueous solution for a specified period of time. When the immersion period is completed, the cases are removed, washed, and prepared for inspection by volatilization of the mercury from the cases. The cases are examined with the aid of a 10× to 20× microscope and the percentage of cracked cases reported.

Most of the investigators have studied the mercury test by determining the time required for cracks to develop in single samples. Ordnance inspection uses the mercury cracking test from the aspect of determining the percentage of failures in a definite number of samples after a fixed period of immersion. It has been necessary to correlate this information with time-to-crack results.

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[*] The presentation of this paper at the annual meeting was sponsored by Committee B-5 on Copper and Copper Alloys, Cast and Wrought.

[1] Junior Metallurgical Engineer and Assistant Metallurgist, respectively, Ordnance Laboratory, Frankford Arsenal, Philadelphia, Pa.

[2] W. H. Hatfield and G. L. Thirkell, "Season Cracking," *Journal*, Inst. Metals, Vol. 22, pp. 67–91 (1919).

Previous work by Gisser[3] on developing a method of replenishing the 1 per cent mercurous nitrate - 1 per cent nitric acid solution after one use, has been extended for the replenishing of 10 per cent mercurous nitrate - 1 per cent nitric acid solutions.

MATERIALS

The investigation of percentage of cases to crack in a given period of immersion was conducted on caliber .30 cartridge cases, manufactured in 1939, by a five-draw process which had all the operations completed except that the primer hole had not been vented, the neck had not been annealed, and the stress relief anneal had not been applied. These cases contained residual stresses of a magnitude which produced different percentages of cracked cases as the cracking power of the mercury solution was changed.

The time-to-crack phase of this investigation was conducted on caliber .50 first-draw pieces which were manufactured in 1940 and had not been washed or annealed. These draw pieces were made with the same punch and die, from a single lot of metal, and in the unannealed state they contained residual stresses of high magnitude.

The composition of both types of samples conformed to the U. S. Army Specification for Cups, Case; Cartridge Brass, No. 57-172-2A.

In making the mercurous nitrate solutions, $HgNO_3 \cdot H_2O$ crystals were used, allowing 6.5 per cent of the gross weight as water of crystallization. The crystals were dissolved in water previously acidified with the required amounts of nitric acid. Solution of the crystals was obtained by gentle heating. The solutions were stored in the presence of small additions of metallic mercury in order to reduce any mercuric salts that might have formed.[4]

In all instances, the percentage of mercurous nitrate was expressed as the number of grams of mercurous nitrate per 100 ml. of solution.[5]

All concentrations of diluted acids referred to in this paper are expressed as percentages by volume of concentrated acid.

The sulfuric acid used in the preparation of samples prior to immersion in the mercurous nitrate solutions had a specific gravity of 1.84; while the phosphoric acid used in pickling had a specific gravity of 1.689.

TESTING PROCEDURES

Time to Crack:

The time-to-crack tests were conducted by immersing the caliber .50 draw pieces in 95 ml. of test solution contained in a 150-ml. beaker. Since the beaker was on a small turntable rotated by hand, all sides of the test specimen could be watched and the time for the first crack to appear noted. Time for the draw pieces to crack was measured by a 10-sec. sweep electric stop clock which could be read to tenths of a second.

The effect of different cleaning methods and different pickling solutions on the time to crack was determined by measuring the time for the first crack to appear after immersion of the prepared draw pieces in 10 per cent mercurous nitrate - 10 per cent nitric acid solutions.

After the draw pieces had been prepared by a standard method of cleaning, the effect of various pickling periods in

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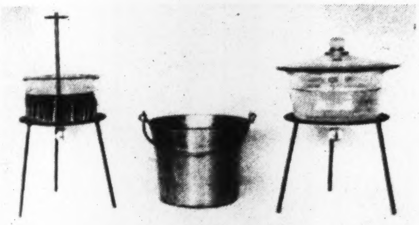
[3] H. Gisser, "A Procedure for Replenishing Spent Mercurous Nitrate Solutions Used in Mercury Cracking Tests," *Report No. 53*, Frankford Arsenal Ordnance Laboratory (1940). This report has not yet been published, but copies may be obtained by applying to the Commanding Officer at Frankford Arsenal.

[4] J. W. Mellor, "A Comprehensive Treatise on Inorganic and Theoretical Chemistry," Vol. IV, pp. 984-997, Longmans, Green and Co., Ltd. (1929).

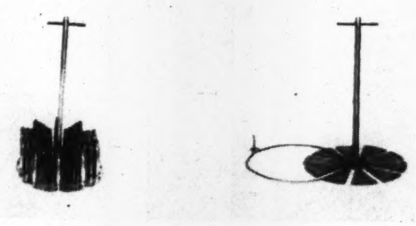
[5] See the requirements for the mercurous nitrate test in the specifications of the American Society for Testing Materials, for example, the Tentative Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part I, p. 396.

40 per cent nitric acid on the time to crack was determined. The standard method of cleaning was as follows: Dip for 5 min. in carbon tetrachloride, rinse in alcohol, wash in water, dip for 5 min. in 12 per cent sulfuric acid, and follow with a water rinse.

The effect of nitric acid and mercurous nitrate concentrations was studied by preparing the draw pieces by the standard method, pickling 60 sec. in 300 ml. of 40 per cent HNO_3, and immersing them in the various test solutions. The time to crack was measured.



Loaded Specimen Holder in H_gNO_3 Solution Water Rinse 42% Nitric Acid Bath



Stainless Steel Holder Loaded with Test Specimens Stainless Steel Specimen Holder

FIG. 1.—Apparatus for Determination of Percentage to Crack.

Percentage to Crack:

The tests conducted with the caliber .30 cartridge cases were made on lots of 50 cases at a time. The cases were held in a definite arrangement by a special fixture made of stainless steel (see Fig. 1).

Pickling was done in 3000 ml. of 40 per cent nitric acid for 30 sec.; immediately after pickling the specimens were vigorously rinsed in water and placed in 2500 ml. of the test solution with the least possible delay. On completion of the immersion period, the cases were removed from the solution, rinsed in water, wiped, and placed on a hot plate for volatilization. Examination for cracks took place immediately after volatilization and air-cooling. This examination of the cases was done with a 20× wide-field binocular microscope.

For cases tested in solutions containing more than 1 per cent mercurous nitrate, it was found necessary to shake the excess mercury off the cases before volatilization; otherwise, considerable difficulty was experienced in volatilizing the mercury. It was also necessary to wipe off the scum which accumulated on the cases during the test to prevent discoloration of the cases during volatilization.

Titration for percentage of mercurous nitrate was carried on by diluting a given volume of sample, acidifying with nitric acid, oxidizing the mercurous ions to the mercuric state with an excess of potassium permanganate, reducing the excess with ferrous sulfate, and titrating to a faint red color with standardized potassium thiocyanate.

Replenishment of 1 per cent mercurous nitrate solutions was conducted in accordance with the procedure outlined in Frankford Arsenal Laboratory Report No. 53.[3] To replenish the 10 per cent mercurous nitrate solution, a 50 per cent mercurous nitrate - 5 per cent nitric acid solution was used and the number of milliliters of this solution to use per liter of spent solution was obtained from a modification of the formula developed in report No. 53.[3]

DISCUSSION OF DATA

The necessity of preparing test specimens prior to immersion in mercurous nitrate solutions has been controversial.

In order to determine whether the pickle of 30 sec. in 40 per cent nitric acid as required by U. S. Army specifications was warranted, the tests shown in Table I were conducted. These results show that a requirement of a pickle is justified. It is sometimes agreed that samples should be tested in the mercurous nitrate solutions in the condition in which they are to be placed in service. This method is not acceptable when it is realized that films that are impervious to solutions are not necessarily impervious to gases.

TABLE I.—EFFECT OF SPECIMEN PREPARATION.
Time in Seconds to Crack in 10 per cent $HgNO_3$ - 10 per cent HNO_3 Solution.

Preparation of Draw Pieces	No Treatment	5-min. naphtha dip Alcohol rinse Water rinse 5-min., 12 per cent H_2SO_4 dip Water rinse		5-min. carbon tetrachloride dip Alcohol rinse Water rinse 5-min., 12 per cent H_2SO_4 dip Water rinse		2-min. dip in 75 per cent H_3PO_4 — 25 per cent HNO_3	60-sec. dip in 40 per cent HNO_3
			30-sec. dip in 40 per cent HNO_3	30-sec. dip in 40 per cent HNO_3	60-sec. dip in 40 per cent HNO_3		
Number of tests	5	10	10	10	100	72	30
Average	no cracks after 900 sec.	163.4	16.9	12.9	8.7	14.8	11.8
Median		135.9	16.4	8.5	7.7	13.4	12.8
Maximum		340.2	37.3	30.9	28.3	38.7	29.7
Minimum		68.5	7.0	3.3	3.8	6.4	4.5
Coefficient of variation, per cent	....	55	48	64	43	47	49

Since the data in Table I reveal no great advantage in using other pickling solutions, it is desirable to use a nitric acid pickling solution to avoid contamination of the test solution by a carry-over from the pickling solution.

The information contained in Table II shows a variation of cracking time with the time of pickle. This indicates the necessity for controlling the pickling time to avoid inconsistent results caused by removing surface metal layers in which high stresses may be present. Experience has shown that a 30-sec. still pickle is sufficient to remove ordinary surface films without appreciably attacking the basis metal, provided no grease or oil is on the surface of the metal. It is necessary to degrease before pickling if oil

TABLE II.—EFFECT OF PICKLING PERIOD.
Time to Crack in Seconds in 10 per cent $HgNO_3$ - 10 per cent HNO_3 Solutions.

Seconds in 40 per cent HNO_3 Pickle	Test No. 1	Test No. 2	Test No. 3	Average
5	61.3	52.0	69.5	60.9
10	28.0	31.9	43.2	34.4
20	18.1	18.8	20.2	18.9
30	....	21.9	20.1	21.0
40	19.6	18.5	17.3	18.5
60	17.6	15.9	17.4	17.0
80	17.9	14.1	14.1	15.4
160	9.7	9.3	9.6	9.5
320	7.7	12.9	7.6	9.4
640	7.2	6.6	9.2	7.7
1200	1.6	1.6	2.0	1.7
1280	4.4	3.6	3.7	3.9
2400	4.2	5.9	12.1	7.4
3600	11.5	11.9	8.6	10.7
4200	17.2	23.2	10.8	17.1

TABLE III.—EFFECT OF $HgNO_3$ AND HNO_3 CONCENTRATIONS.
Time to Crack in Seconds in $HgNO_3$ Solutions Using Standard Cleaning Method and 60-sec. HNO_3 Pickle.

Number of pieces tested.	23	23	23	100	23	23
HNO_3 concentration, per cent	1	10	1	10	1[a]	10
$HgNO_3$ concentration, per cent	1	1	10	10	20	20
Average	40.4	55.2	7.1	8.7	13.8	9.9
Median	35.6	48.6	6.5	7.7	11.7	8.4
Maximum	79.9	109.2	16.6	28.3	26.4	21.6
Minimum	25.3	27.6	4.0	3.8	4.8	4.0

REMARKS.—Thick coating of mercury and bubbles made cracks hard to detect in the 20 per cent mercurous nitrate solutions.

[a] Acid was added to keep the salts from crystallizing out; approximate concentration was 3 per cent.

and grease are present, since pickling is seriously retarded and erratic results are obtained.

It has been stated in the literature that nitric acid concentration, and not mercury salt concentration, is the variable which determines the cracking power of mercury salt solutions.[6]

TABLE IV.—THE EFFECT OF $HgNO_3$ AND HNO_3 CONCENTRATION IN THE TEST SOLUTION.
15-min. Immersion Period.

Lot	$HgNO_3$, per cent	HNO_3, per cent	Number of Samples	Percentage of Specimens Cracked
No. 1	1.0	0.1	50	16
No. 2	1.0	1.0	50	20
No. 3	1.0	10	50	16
No. 4	1.0	20	50	20
No. 5	10	1.0	50	36
No. 6	10	10	50	34
No. 7	10	20	50	30

TABLE V.—THE EFFECT OF DEPLETION OF $HgNO_3$ ON CRACKING POWER OF TEST SOLUTIONS.
15-min. Immersion Period.

Lot	Series I		Series II		Series III		Series IV		Series V		Average of Series I to V		
	C^a	H^b	C^a	H^b	C^a	H^b	C^a	H^b	C^a	H^b	C^a	H^b	Percentage $HgNO_3$ Depleted
No. 1	30	..	22	10.3	32	9.9	32	9.8	40	9.7	31.2	9.9	4.0
No. 2	28	..	24	6.7	26	6.0	32	5.9	40	5.0	30.0	5.9	3.4
No. 3	24	..	26	2.2	12	2.9	24	2.2	18	2.7	20.8	2.5	1.0
No. 4	30	..	24	1.1	20	1.7	22	1.4	20	1.9	23.3	1.5	..

[a] C = Percentage specimens cracked.
[b] H = Percentage $HgNO_3$.

Tables III and IV show that the mercury salt concentration of a solution, and not the nitric acid concentration, is the influential component. The 10 per cent mercurous nitrate solution has a much greater cracking power than the 1 per cent mercurous nitrate solution. The investigators who found that increased acid concentration caused an increase in the cracking power of the test solution evidently did not pickle their specimens prior to testing. Therefore, an apparent increase in cracking power is to be expected in view of the fact that high acid concentration solutions would tend to be more self-pickling.

Cracking of the cases invariably took place just above the extractor groove (K position), which simplified the examination for cracks. Since the necks had not been annealed, this part of the case cracked 100 per cent in all lots. All references to cracks found refer to those located in the K position.

The results in Table V show that a 10 per cent mercurous nitrate solution loses some of its cracking power when its mercury concentration has dropped below approximately five per cent. In order to be sure the mercury concentration is always above this percentage, a solution should be replenished after each use. If samples of small surface area are to be tested, small volumes of solution can be used and the spent solution saved until sufficient quantities are on hand to warrant the time to titrate and replenish the solution. All stored solutions should contain metallic mercury in order that the solutions remain in the mercurous state.

The information contained in Table VI and Fig. 2 shows that 10 per cent mercurous nitrate solutions may be re-used up to at least 13 times if the solution is replenished after each use by the method outlined in this paper.[7] The re-use of replenished 1 per cent solutions has been investigated by Gisser.[3] He states that 1 per cent mercurous nitrate solutions may be replenished and re-used until the copper content increases to 0.25 per cent, at which time the copper interferes with the titration method used in replenishment of the solution.

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[6] J. D. Jevons, "The Metallurgy of Deep Drawing and Pressing," John Wiley and Sons, New York, N. Y. (1940).

[7] See p. 899.

An analysis of the used 10 per cent mercurous nitrate solution after the thirteenth lot of cases had been tested in the solution showed that the copper and zinc replacement of the mercury in solution was of the ratio of 70 parts of copper to 30 parts of zinc. Calculations of the amount of mercury depleted from the solution in 13 tests and the amount of copper and zinc in the solution showed that the copper and zinc were ionized to the "ic" state and replaced two atoms of mercury for each atom of copper and zinc dissolved into the solution.

Table VII presents information on the cracking power of 1 per cent mercurous nitrate solutions for given immersion periods. From this table it can be seen that a 60-min. period of immersion in a 1 per cent solution is more searching than

TABLE VI.—EFFECT OF COPPER AND ZINC ACCUMULATION IN REPLENISHED TEST SOLUTION.

15-min. Immersion Period.

Lot	Number of Specimens	Percentage $HgNO_3$		Percentage Cracked		
		X Series	*Y* Series	*X* Series	*Y* Series	Average
No. 1	50	9.6	9.9	26	26	26
No. 2	50	10.4	10.4	20	30	25
No. 3	50	10.4	10.0	20	24	22
No. 4	50	9.9	9.6	32	26	29
No. 5	50	10.0	9.7	22	22	22
No. 6	50	10.0	10.2	28	20	24
No. 7	50	10.0	10.0	26	34	30
No. 8	50	9.9	9.4	28	24	26
No. 9	50	10.0	10.2	34	26	30
No. 10	50	10.4	10.0	32	28	30
No. 11	50	9.7	10.2	26	20	23
No. 12	50	10.0	10.4	20	22	21
No. 13	50	10.1	10.1	32	20	26

TABLE VII.—EFFECT OF TIME OF IMMERSION ON CRACKING POWER OF TEST SOLUTION.

Lot	Number of Specimens	Immersion Period, min.	Percentage Cracked	Percentage $HgNO_3$	Percentage $HgNO_3$ Depleted
No. 1	50	15	16	1.0	...
No. 2	50	15	20	1.0	...
No. 3	50	15	16	1.0	...
No. 4	50	15	20	1.0	...
Average			18.0	1.0[a]	...
No. 1	50	30	22	1.1	...
No. 2	50	30	30	1.1	...
No. 3	50	30	24	1.2	0.4
Average			25.3	1.1	0.4
No. 1	50	60	34	1.3	0.6
No. 2	50	60	36	1.1	0.5
No. 3	50	60	34	1.2	0.6
Average			34.7	1.2	0.6
No. 1	50	60	36	1.1	0.5
No. 2	50	60	30[b]	1.2[b]	0.6
No. 3	50	60	38[b]	1.1[b]	0.5
Average			34.7	1.1	0.5

[a] Nominal per cent.
[b] Replenished solution.

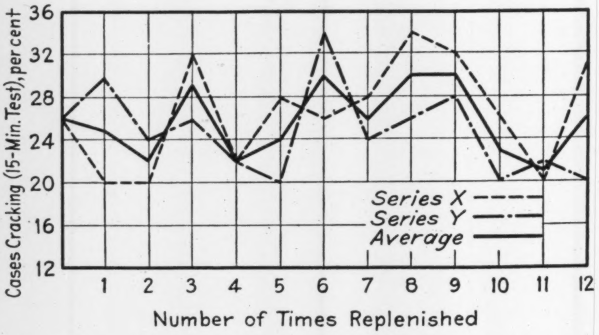

FIG. 2.—Effect of Re-Use of Replenished 10 per cent $HgNO_3$ Solution on Cracking Power.

a 15-min. immersion in a 10 per cent solution.

Table VII also shows that a longer period of immersion is more consistent than a shorter period test. This can best be seen by comparing the results of first run lots in Table IV with the results of the 60-min. tests in Table VII. The percentage of cracked cases in the test with 15-min. immersion in 10 per cent mercurous nitrate solution varied from 22 to 40, while in the test with 60-min. immersion in 1 per cent mercurous nitrate solution the percentage of cracked cases varied from 30 to 38. Since it is an accepted fact that long-time tests are more consistent than short-time tests, these results are more or less to be expected.

TABLE VIII.—EFFECT OF TEMPERATURE ON CRACKING POWER OF TEST SOLUTION.
60-min. Immersion Period.

Lot	Number of Samples	Temperature of Solution, deg. Fahr.	Percentage Cracked	Percentage $HgNO_3$	Percentage $HgNO_3$ Depleted
No. 1	50	34	28	1.2	0.5
No. 2	50	34	22	1.0	0.4
No. 3	50	34	28	1.0	0.4
Average			26	1.1	0.4
No. 1	50	70	34	1.3	0.6
No. 2	50	70	36	1.1	0.5
No. 3	50	70	34	1.2	0.6
Average			34.7	1.2	0.6
No. 1	50	110	20	1.1	0.9
No. 2	50	110	24	1.3	0.9
No. 3	50	110	22	1.2	0.8
Average			22	1.2	0.9

The results shown in Table VIII do not agree with the results of certain other investigators. Mr. Croft[8] states that as the temperature of the mercurous nitrate solution increases, the cracking power of the solution increases. From data in Table VIII, it appears that there is a maximum cracking power at about room temperature (70 F.). The effect of temperature was investigated on 1 per cent solutions for a 60-min. period of immersion. This test was used as it gave the most consistent results of all the tests investigated in this paper. The temperatures were held constant by using an ice bath for the 34 F. test and a thermostatically controlled electric oven for the 110 F. test. The temperature of the ice solution did not vary more than 4 deg., while the temperature of the oven was held at 110 ± 3 F.

The effect of time the mercury remains on the specimens after the test period was completed was investigated by Murdza.[9] He used the percentage of cases to crack after a 15-min. immersion period in 1 per cent mercurous nitrate solution and subsequent lapse of time before inspection. Since these data were in agreement with experience acquired during routine testing at Frankford Arsenal, duplicate tests were not conducted.

All cases were volatilized free of mercury in order to avoid overlooking any fine cracks which otherwise might be bridged by the mercury coating. Experience at Frankford Arsenal has shown that volatilization of mercury is necessary to inspect ordnance materials.

CONCLUSIONS

From the information accumulated in this investigation the following may be concluded.

1. When testing copper-base alloys for excessive stresses by the mercurous nitrate solution method, a standard preparation of the sample should be required. A good surface conditioner such as a 40

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[8] Private communication from H. P. Croft, Chief Metallurgist, Cleveland Plant, Chase Brass and Copper Co.

[9] N. H. Murdza, "Study of Some Variables Affecting the Results of Mercury Cracking Test as Applied to Small Arms Cartridge Cases," *Report No. 32B*, Frankford Arsenal Ordnance Laboratory (1940). This report has not yet been published, but copies may be obtained by applying to the Commanding Officer at Frankford Arsenal.

per cent nitric acid pickle for 30-sec. should be specified.

2. The mercurous nitrate solutions should be made from crystals which have a known water of crystallization so that allowances can be made for its weight. The crystals should be dissolved in dilute aqueous acid solutions with as little heating as possible. Precaution should also be taken to reduce any oxidized salts to the mercurous state by storing the solution in the presence of metallic mercury for at least 24 hr.

3. Immersion for 60 min. in 1 per cent mercurous nitrate solution should be specified unless it is necessary to obtain results in a shorter length of time. Use of high mercurous nitrate concentrations for shorter periods to obtain equivalent cracking power can only be justified when time is at a premium.

4. The re-use of replenished 1 per cent mercurous nitrate solutions is allowable, provided the copper concentration is lower than 0.25 per cent. The method developed at Frankford Arsenal is a very easy method of replenishing spent solution.

5. The acid concentration of the solution is not important except that it should be limited to such an extent that it will not react with the specimens or the mercury coating.

6. The ratio of solution volume to surface area exposed should be at least in the order of 10 ml. of solution to 1 sq. in. of surface area exposed to the solution.

7. The solution should be maintained at room temperature (70 ± 10 F.).

8. Specimens should be volatilized free of mercury, prior to inspection, at a temperature not exceeding 500 F. Samples should be washed to remove scum so that there is as little discoloration of the specimens as possible.

9. The specimens should be examined with the aid of a ×10 to 20 magnifying glass or microscope.

Acknowledgment:

It is the authors' wish to express their appreciation to Major L. S. Fletcher, Frankford Arsenal; Major H. P. Croft, O. R. C. Chase Brass and Copper Co.; and R. D. France, Frankford Arsenal, for their suggestions and advice in conducting this investigation.

NOTE.—An extensive bibliography has been prepared to be appended to this paper but it has not been found possible to include it in the preprint. However, the bibliography will appear with the paper as finally published.

[See Bibliography on Season Cracking, p. 918.–ED.]
[See Joint Discussion, p. 927.–ED.]

INFLUENCE OF EXTERNAL STRESSES ON TENDENCY OF BRASS WIRES TO STRESS-CORROSION CRACK, AS INDICATED BY THE MERCUROUS NITRATE TEST[1]

By Harry P. Croft[2]

Synopsis

This paper shows that the tendency of brass wire to stress-corrosion crack, as indicated by the mercurous nitrate test, depends not only upon the degree, but also on the type of cold work, and that this tendency attains a peak value at a certain degree of cold reduction in drawn wires, which phenomenon is not paralleled by wires that have been cold-worked by stretching. The fact is also demonstrated that the accelerating influence of increasing grain size on the tendency to stress-corrosion crack, previously shown by Morris[3] as important in externally stressed annealed metal, actually continues to be a strong factor in influencing time to crack in metal that has been cold-worked by drawing or stretching after annealing. This influence continues to have such a tendency even in wires that have been cold-drawn and subsequently given a low-temperature or "relief" anneal after the drawing operation.

This work on stress-corrosion cracking of brass was undertaken at the request of the Ordnance Department of the United States Army, as it had been found that some ordnance material was subject to stress-corrosion cracking after being kept in storage for extended periods. This portion of the results is submitted before the completion of the investigation because it is felt that this information could be used at the present time in the defense program.

There has been a vast amount of work carried out on the subject, some two hundred references having been cited in a bibliography prepared at Frankford Arsenal.[4]

Since the exact time of cracking is often difficult to observe in samples that are merely internally stressed and tested in the standard mercurous nitrate solution, because of screening of the cracks by a deposited layer of mercury, and since considerable scattering of results is found in any such series of tests made on samples prepared on a commercial basis, due to slight unnoticeable irregularities, it was decided to use a method of imposing external loads on brasses of various characteristics so as actually to fracture the specimens to indicate definitely the time of cracking.

Round wires were selected for the first

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[1] Portion of a dissertation which will be presented to the Faculty of Case School of Applied Science in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

The presentation of this paper at the annual meeting was sponsored by Committee B-5 on Copper and Copper Alloys, Cast and Wrought.

[2] Assistant Director of Research, Cleveland Plant, Chase Brass and Copper Co., Cleveland, Ohio.

[3] A. Morris, "Stress-Corrosion Cracking of Annealed Brasses," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., p. 256 (1930).

[4] H. Rosenthal and A. L. Jamieson "Mercury Cracking Test, Procedure and Control," see p. 897.

part of the investigation, in order to minimize the number of variables by having only one dimension involved. These wires were held in tension under predetermined loads while being exposed to the corroding media.

EQUIPMENT AND MATERIALS

The apparatus employed (Fig. 1) was a modification of that used by Morris,[3] which has been employed by Crampton and his associates in the main laboratory of the Chase Brass and Copper Co. at Waterbury, Conn., for other unpublished investigations. This equipment consisted of three units, each having a lever arm capable of holding the wire in tension under any desired loads, which were obtained by means of 10-lb. weights added at the end of the arm. Ratios of 8 to 1 and 10 to 1 were obtainable by changing the location of the load, and a sliding weight on the lever allowed for application of intermediate loads.

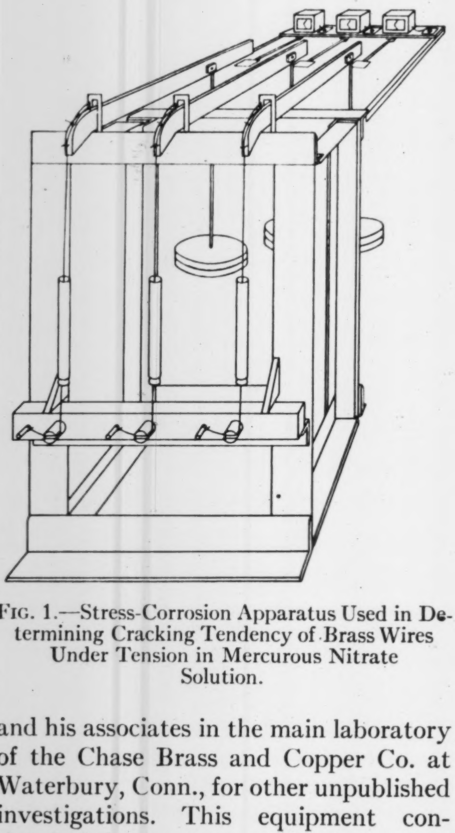

FIG. 1.—Stress-Corrosion Apparatus Used in Determining Cracking Tendency of Brass Wires Under Tension in Mercurous Nitrate Solution.

The solution was held in contact with the wires by means of bakelite tubes $\frac{3}{4}$ in. in inside diameter and 8 in. long, seated vertically around the wires on unused rubber stoppers through which the wires had previously been threaded.

Timing of the cracking period was carried out with electric clocks, located at the ends of the lever arms. As the samples broke, releasing the levers, the switches, which were located in the paths of the ends of the arms, were opened, stopping the clocks. In addition to this, an electric timer with a ten-second sweep, readable to tenths of a second, was used for samples which broke on short exposures. This timer was started and stopped by a finger switch.

At the time that this investigation was undertaken, considerable discussion had arisen concerning the merits of the mercurous nitrate test as compared to that of ammonia vapor. Since the control of concentration of the nitrate solution is more easily maintained, it was decided to carry out the first part of the investigation using this medium. Accordingly, a solution containing 10 per cent mercurous nitrate and one per cent nitric acid was employed, because, due to preliminary work at Frankford Arsenal by A. Jamieson and the author, it had been decided that this solution would be standard for future tests. The exact composition was:

$HgNO_3 \cdot H_2O$	121 g.
HNO_3 (sp. gr. 1.42)	11.2 ml.
H_2O	1000 ml.

Cartridge brass wire of 0.109-in. diameter was employed for the tests. One series also was run using 65–35 common high brass wire in which no beta constituent was visible under microscopic examination. The common high brass series was merely used for a check, and

TABLE I.—COMPOSITION OF WIRES.

	Cartridge Brass	High Brass
Copper, per cent	70.04	66.02
Lead, per cent	0.01	0.01
Iron, per cent	0.01	0.01
Zinc, per cent	29.94[a]	33.96[a]

[a] By difference.

the results were found to be within the range of the 70-30 wires tested under the same conditions. The exact compositions are given in Table I and the tensile strengths in Table II.

The soft wires were prepared by drawing the wires six B. & S. numbers hard to the finish size and annealing them in a convection type air-heated furnace, with the coils of wire directly over the thermocouple. Relief annealing was carried out in a laboratory size Lindbergh Cyclone Furnace.

The cold-stretched wires were also reduced six B. & S. numbers hard to the ready-to-finish size, at which time after annealing they were given a flash pickle in cold 10 per cent sulfuric acid, rinsed, and pulled on a wire testing machine the amount required to obtain the desired temper in a diameter of 0.109 in. The stressed wires, after having been pulled to predetermined diameters, were fastened to supports and cut from the jaws of the testing machine. It was also necessary to apply the loads gently in order to avoid strains in excess of those desired. For check purposes, one series was given a flash dichromate dip, after pickling, in a solution containing 0.25 lb. of sodium dichromate per gallon of 10 per cent sulfuric acid.

The cold-drawn material was also reduced six B. & S. numbers hard to the ready-to-finish size, annealed, pickled, and drawn to the finish size through a tungsten carbide die having an included angle of 30 deg. All wires were wrapped as soon as processed and kept in this condition until tested. To minimize oxidation, they were tested as soon as possible after fabricating. It had been determined that no appreciable relief or redistribution of stresses took place upon standing, but the amount of oxide does

TABLE II.—MECHANICAL PROPERTIES OF BRASS WIRE.

Nominal Temper, B. & S. Numbers	Diameter, in.	Grain Size, mm.	Tensile Strength, psi.	
			Drawn	Stretched
Soft	0.109	0.140	45 000	....
Soft	0.109	0.070	44 900	....
Soft	0.109	0.020	50 100	....
¼	0.109	0.140	52 500	50 100
½	0.109	0.140	54 100	54 000
¾	0.109	0.140	58 900	56 300
1	0.109	0.140	64 300	59 600
1½	0.109	0.140	69 700	62 200
2	0.109	0.140	80 000	....
4	0.109	0.140	108 600	....
8	0.109	0.140	131 900	....
1	0.097	0.140	....	64 200
1	0.097	0.070	....	65 500
1	0.097	0.020	....	72 000
2	0.101	0.090	75 000	....
2	0.101	0.020	82 500	....
2—Relief annealed	0.101	0.090	73 700	....
2—Relief annealed	0.101	0.020	83 100	....
1	0.109	0.040	64 100	....
1—Relief annealed, deg. Fahr. 350	0.109	0.040	64 200	....
400	0.109	0.040	66 900	....
450	0.109	0.040	63 700	....
500	0.109	0.040	63 200	....
550	0.109	0.040	63 200	....
600	0.109	0.040	61 600	....
650	0.109	0.040	60 500	....
700	0.109	partially recrystallized	54 100	....

increase, and due to the fact that any removal of metal by pickling of the finished wire containing internal stresses redistributes these stresses, it was desirable to test these wires without additional pickling. The cold-drawn wires were prepared for testing by cleaning with liquid ether, followed by vapor degreasing with ether gas.

Temperature control was found by preliminary experiments to be very important. The results of some of these tests are given in our comments on Rosenthal and Jamieson's paper.[5] We consequently controlled the temperature of the room at 70 ± 5 F., and almost all of these tests were carried out at a temperature of 70 ± 2 F.

TABLE III.—CRACKING TENDENCY IN MERCUROUS NITRATE SOLUTION OF BRASS WIRE HARDENED BY COLD STRETCHING.

Wire Diameter, 0.109 in.

External Load, psi.	Cracking Time, sec.					
Approximate temper, B. & S. Nos.	Soft Annealed	¼	½	¾	1	1½
Reduction of area by stretching, per cent		5	8.5	16	21	28
20 000	4 980	22 400	31 500	82 400	19 900	72 000
23 000	....	3 860	9 400	14 200	14 800	29 800
28 500	12.3	131	779	813	968	2 440
33 500	....	....	23.6	82.3	117	168
37 000	....	....	5.2	26.9	83.9	431
45 000	....	....	....	6.0	11.9	17.5
55 000	....	....	....	....	....	3.9

It was found necessary to have the wires absolutely free from any minute scratches or kinks, as the presence of such stress raisers influenced the time of cracking considerably.

TEST METHODS

The load was applied by passing the wire through a split drum, which was revolved to produce the necessary tension and then locked in place by a nut on the end of the shaft. Thirty milliliters of the mercurous nitrate solution, at room temperature, were introduced as rapidly as possible into the bakelite tube, and the timer was started simultaneously.

Some wires loaded beyond their yield strengths stretched during the loading. As was to be expected, such stretched wires cracked in shorter periods than those which were subjected to lighter loads. Distinct creep was noticed during the testing of some annealed and some slightly hardened wires, the amount depending upon the external load as well as the original temper.

Some wires necked down from a fraction of a second to a few seconds before breaking. The drawn wires, however, broke sharply. The fractures of the test specimens occurred anywhere from the junction of the wire with the rubber stopper to the "water line," but the majority failed between these extremes.

DISCUSSION OF DATA

In tests such as these, there are three or more effects taking place simultaneously:

1. The solution of an outside layer of the wire, the amount going into solution depending upon the time before failure. This was checked by diameter measurements of some wires which lost several thousandths of an inch under exposures of the order of one or more weeks.

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[5] See p. 897.

2. Formation of a mercury amalgam. The formation of an amalgam of appreciable thickness was indicated by tests of unstressed samples coated with mercurous nitrate solution for various periods, then frozen with liquid air, and pulled in a tension testing machine while still frozen. Since the physical properties decreased slightly in proportion to the time of exposure, it is believed that such an amalgamation occurred.

3. The cracking leading to rupture. Failure occurred in these tests when, due to the combined effect of these three factors, the externally stressed cross-sectional area was reduced to such a size that its tensile strength was equalled by the constant external load. This often occurred immediately after the yield strength was exceeded. Since the first two effects were of small importance in testing specimens which failed in a short time, and since this importance increased with the time of the test, the results have been plotted on semilogarithmic and reciprocal-square scale charts to emphasize the significance of the short-time failures.

The effect of the degree of cold working on the cracking tendency of stretched wires is shown in Fig. 2. The results for this series of tests are given in Table III. The fact that the wire would not stretch beyond 28 per cent without fracturing prevented the higher values from being investigated. Up to this point, the greater the amount of cold work, the lower is the tendency to crack. The cracking tendency increases rapidly with increase of load. While wires that are hardened by cold stretching might be considered to be stressed uniformly throughout, this is not strictly true, as the outside grains are only partially supported during the stretching, while internal grains are of course supported on all sides. The data illustrated in Fig. 2 have been replotted in Fig. 3 as a function of the external load, each curve showing the cracking tendency for a given degree of cold work. Below about 15,000 psi.

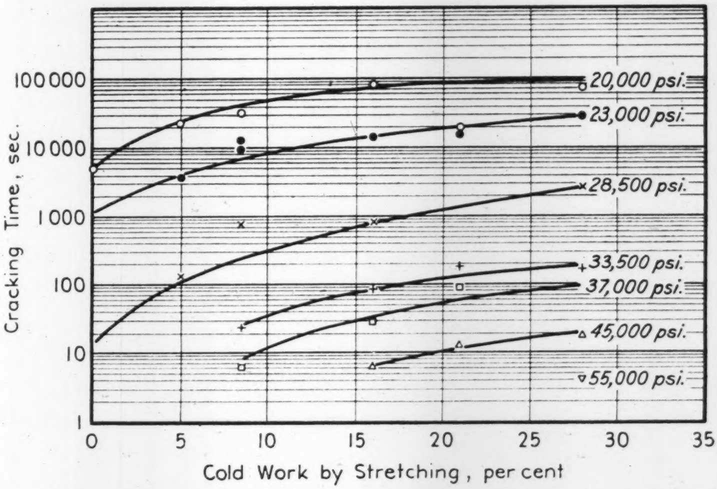

FIG. 2.—Effect of Degree of Cold Work on Cracking Tendency in Mercurous Nitrate Solution of Cold-Stretched Brass Wire with Various External Loads.

Wire diameter, 0.109 in.; previous grain size, 0.140 mm.

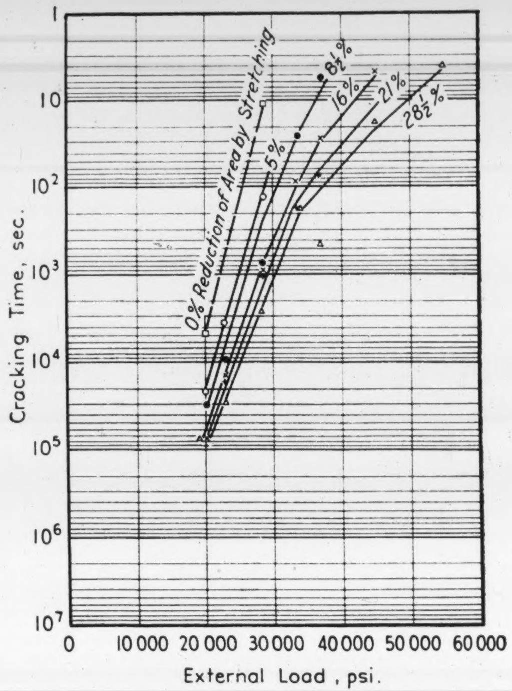

FIG. 3.—Effect of External Loads on Cracking Tendency in Mercurous Nitrate Solution of Cold-Stretched Brass Wire of Various Tempers.

Wire diameter, 0.109 in.; previous grain size, 0.140 mm.

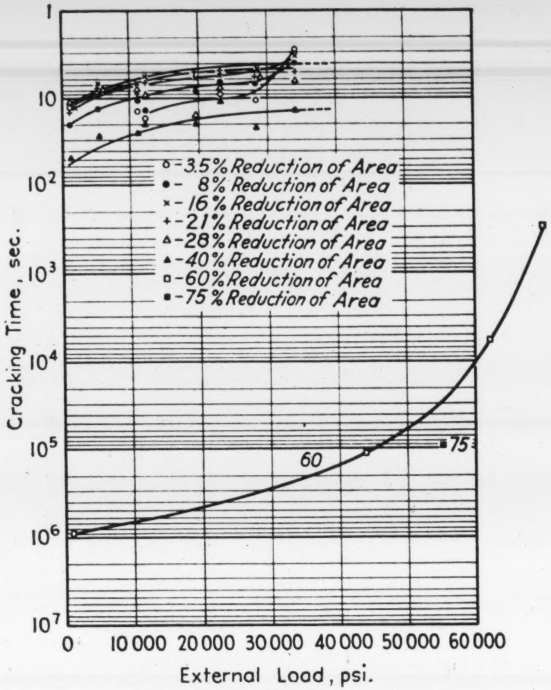

FIG. 4.—Effect of External Loads on Cracking Tendency in Mercurous Nitrate Solution of Cold Drawn Brass Wire of Various Tempers.

Wire diameter, 0.109 in.; previous grain size, 0.140 mm.

external load, the tendency to crack appears to approach zero, as extrapolation of these curves indicates cracking periods of the order of a year.

The results on the wires which were pickled before cold-stretching were duplicated by tests made on wires which were pickled and given a flash dichromate dip. This part of the method of preparation was evidently unimportant, as any difference in the results was indistinguishable.

The effect of similar loads on the cracking tendency of drawn wires finished with approximately the same reductions as the stretched wires is shown in Table IV and Fig. 4. Here the cracking tendency for reductions between 3.5 and 40 per cent is uniformly high for low external loads and increases but slightly with additional loading. With external loads exceeding the yield points, where some stretching may have taken place, the time of cracking decreases rapidly, the exact effect probably being masked by difficulties of manipulation in carrying out tests where the cracking times were less than 5 sec. The tendency to crack increases with the degree of cold reduction up to the 21 per cent reduction curve and then falls off again, the 40 per cent reduction curve (approximately 2 B. & S. Nos. hard) falling just below that for the 3.5 per cent reduction curve (¼ B. & S. Nos. hardness). These curves appear to have the same trend. By increasing the draw to 60 per cent reduction, (4 B. & S. Nos. hard) a very considerable drop in tendency to crack was experienced. The wires, instead of breaking at right angles to the direction of drawing, fractured at 45 to 60 deg. to that direction and often cracked longitudinally before breaking in tension. This was probably due to the fact that the circumferential stresses exceeded the combined internal and external longitudinal stresses. The tendency of the 8 numbers hard, or spring temper, wire to crack was even less than the 4 numbers hard wire; in fact, with light loads, samples did not fail in over 1,000,000 sec., at which time the tests were terminated.

TABLE IV.—CRACKING TENDENCY IN MERCUROUS NITRATE SOLUTION OF BRASS WIRE HARDENED BY COLD DRAWING.

Wire Diameter, 0.109 in.

External Load, psi.	Cracking Time, sec.							
Approximate temper, B. & S. Nos.	¼	½	¾	1	1½	2	4	8
Reduction of area by drawing, per cent	3.5	8.0	16	21	28	40	60	84
1 070	12.0	18.7	12.0	13.5	10.9	46	943 000	....
5 330	....	12.0	6.6	7.1	7.5	25	....	....
10 900	14.5	11.0	6.7	8.6	8.0	23.8	....	....
12 000	15.1	13.9	5.5	6.0	9.0	18.2	....	....
19 600	....	7.0	5.4	4.9	14.0	19.9	....	....
23 000	8.3	6.2	5.3	4.5	7.4	9.9	....	....
28 200	10.1	6.5	4.4	5.6	5.7	23.4	....	....
33 700	2.5	3.7	3.1	4.9	6.1	12.7	....	....
44 000	....	....	....	....	....	....	109 000	....
55 100	....	....	....	....	....	....	....	93 600
61 900	....	....	....	....	....	....	6 120	....
70 400	....	....	....	....	....	....	289	....

That a critical degree of reduction resulting in a minimum cracking time exists for drawn wire is shown more clearly in Fig. 5, in which values from Figs. 3 and 4 for an external load of 25,000 psi. have been plotted as a function of the degree of cold working. The cracking tendency of the stretched wires, starting with a high value for low reduc-

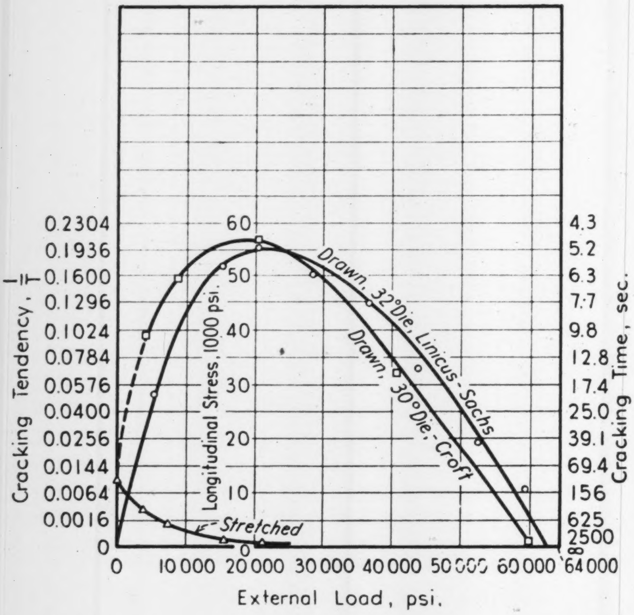

FIG. 5—Effect of Various Amounts of Cold Reduction on Cracking in Mercurous Nitrate of Stretched and Drawn Wires Subjected to 25,000-psi. External Load.

Drawn wire diameter, 0.109 in.; previous grain size, 0.140 mm. Diameter of Linicus-Sachs wire, 0.118 in.

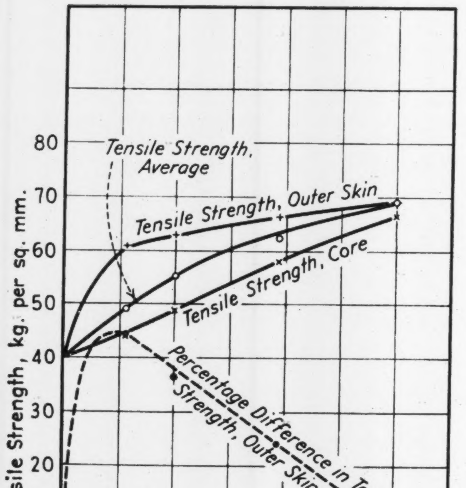

FIG. 6.—Average Tensile Strength of the Core and Outer Skin of 0.118-in. Diameter Drawn Brass Wire as a Function of Percentage of Cold Reduction.

W. Linicus-G. Sachs, Sup. Issue XVI, *Mitteilung Deutsches Materialprüfungsamt* Berlin (1931).

tions, falls off uniformly to the 28 per cent reduction, which was the maximum stretch obtainable by this method.

The curve for drawn wires, on the other hand, shows a medium cracking tendency for light reductions, and this tendency increases with the degree of reduction, reaching a maximum in the neighborhood of 21 per cent reduction and then falling off rapidly to a very low value at the 60 per cent reduction of area. The curve roughly parallels one given by Linicus and Sachs,[6] which has been superimposed on this chart. They located a similar peak in the amount of stress in wires of 0.118-in. diameter, drawn through a 32-deg. angle die. Their method of determining the stress was by holding the wires firmly and shaving off one half of the section, calculating the residual stress from the amount of bending that followed the release of the ends of the wire. It would be expected that the peak in the cracking time curve would coincide with that of the curve for stress determination, and that these curves would parallel each other. Linicus and Sachs' work was done on brass wire containing 63 per cent copper and 37 per cent zinc, and no mention is made of the presence or absence of the beta constituent. They have determined that this residual stress resulted from the difference in the tensile strength between the outer skin and the core of the wire, their results on this determination being shown in Fig. 6. Here again, the curve showing difference in tensile strength parallels roughly the Linicus-Sachs stress curve and the author's cracking time curve for a die of the same included angle, shown in Fig. 5. The area between the curve for the drawn wire and that for the stretched wire in Fig. 5 probably represents the amount of stress resulting from the nonuniform flow through the die.

It is possible that in imposing external loads on the drawn wires, which were only stressed internally, these residual stresses are redistributed in such a manner that the outer fibers do not exceed the yield strength until the entire cross-section attains that value, after which the external loads affect drawn wires in the same way as the stretched wires; that is, above the yield point, the curves might coincide with those of the stretched wires. Although this tendency may be indicated by the results obtained, it is difficult to prove, due to the fact that the yield strength of yellow brass wires of various tempers is a questionable value and the subject of much

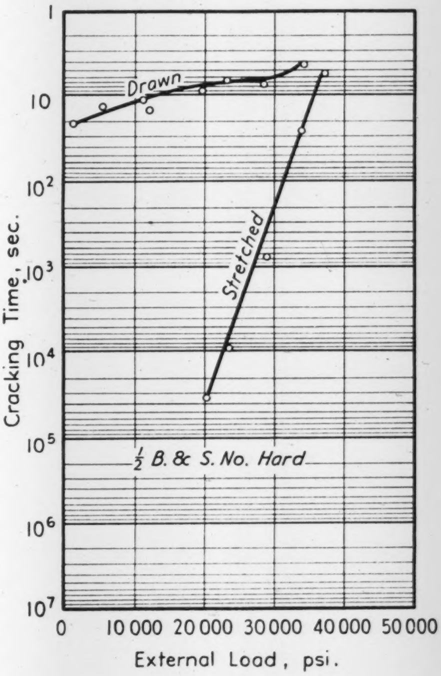

FIG. 7.—Comparison of Cracking Tendencies of Wires Cold-Stretched and Cold-Drawn 8 per cent, Respectively, from 0.140-mm. Anneal.

Wire diameter, 0.109 in.

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[6] W. Linicus and G. Sachs "Experiments on the Properties of Drawn Wires," Supplemental issue of *Mitteilung Deutsches Materialprüfungsamt* (1931); also in G. Sachs "Plastische Verformüng," *Handbuch der Experimentalphysik,* Band 5, Leipzig (1930).

controversy. It is apparent, however, that the cracking tendency curves of the drawn wires meet those for the stretched wires which have been reduced the same amount at certain points (Fig. 7) at which the tendency to crack is quite high and that some regular trend which should be related to some mechanical characteristic of the wire is indicated.

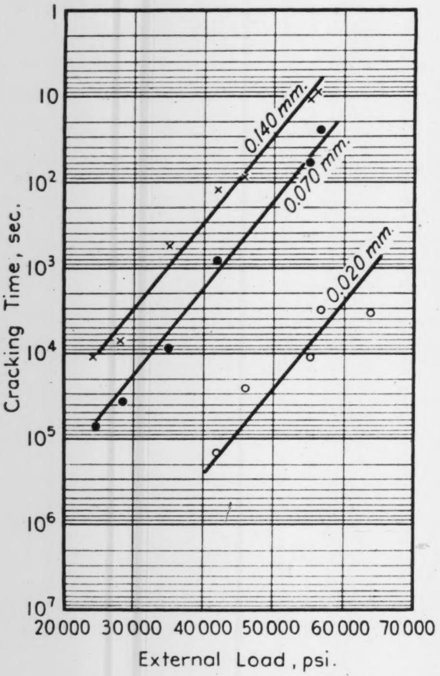

FIG. 8.—Effect of Grain Size on Cracking Tendency in Mercurous Nitrate Solution of Brass Wire Cold-Worked by Stretching.

Reduction of area, 21 per cent; wire diameter, 0.097 in.

Morris[3] showed the effect of grain size on cracking time of annealed brass rods. This influence, as was to be expected, was confirmed for wires of the size and composition investigated by the author. (Figs. 3 and 10.) Since season cracking in alpha brass and other metals has long been known as a grain boundary phenomenon, there was reason to suspect that the influence of grain size was important not only on annealed metals, as shown by Morris, but also on cold-worked metals. Further tests were accordingly made on wires of three different grain sizes which had been cold-stretched various amounts. The results for 0.097-in. wire stretched 21 per cent are given in Table V and Fig. 8. The curves for the externally stressed wire of various grain sizes (shown as straight lines but probably slightly convex) are roughly parallel and show that wires with large grain sizes crack more rapidly than those with smaller grain sizes. Similar results were obtained with wires stretched to produce other degrees of reduction, but with lighter reductions the wire having 0.020 mm. grain size could not be made to crack during reasonable exposures. The effect of grain size on drawn wires given the same reduction and subjected to various external loads is shown in the upper half of Fig. 9, and the results are given in Table VI. Here again, fine grain size wires showed a considerable superiority over coarse in resisting the influence of mercurous nitrate.

TABLE V.—EFFECT OF GRAIN SIZE ON CRACKING TENDENCY OF EXTERNALLY LOADED BRASS WIRE COLD-WORKED BY STRETCHING 21 PER CENT.

Wire Diameter, 0.097 in.

External Load, psi.	Cracking Time, sec.		
Grain size, mm....	0.140	0.070	0.020
24 700	11 640	74 800	....
28 700	7 380	24 700	....
35 500	542	8 708	....
42 200	117	819	143 000
46 300	86	....	26 200
55 800	11.9	60.2	11 800
57 100	8.3	23.8	3 160
64 300	....	....	3 470

It might be expected that the influence of grain size would be eradicated by a low-temperature or relief anneal. This was investigated on wires of various drawn tempers and the results of two different grain sizes, 0.101-in. wire, drawn 35 per cent and relief annealed for 20 min. at 450 F. are given in Table VII, illustrated in the lower half of Fig. 9. Curves are shown for the 2 B. & S. Nos. hard wires finished from 0.090 and 0.020 mm. respectively, and for the same wires after being subjected to the relief annealing temperature. While an improvement in the resistance to cracking by mercurous nitrate is shown in both cases, this was less than had been expected, and the effect of the grain size resulting from the anneal previous to the temper draw is still quite pronounced, the finer grain size material showing a considerably greater immunity to cracking.

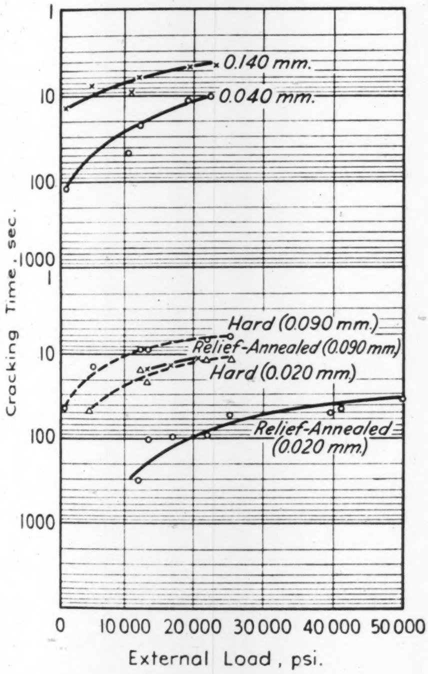

FIG. 9.—Effect of Grain Size on Cracking Tendency in Mercurous Nitrate Solution.

Upper half.—0.109-in. Brass Wire Drawn 21 per cent. *Lower half.*—0.101-in. Brass Wire Drawn 35 per cent and Relief-Annealed at 450 F.

There are three usual methods of counteracting the tendency to season crack of fabricated brass. As mentioned by a previous investigator,[7] these are:

1. The use of an alloy, richer in copper content than the yellow brasses, which is relatively immune to this type of attack.

2. A reduction of the internal stresses to a safe figure by cold working operations which set up opposing stresses; these include straightening, springing, and other forms of deformation.

3. The use of low-temperature or stress "relief" anneals.

TABLE VI.—EFFECT OF GRAIN SIZE ON CRACKING TENDENCY OF EXTERNALLY LOADED BRASS WIRE COLD-WORKED BY DRAWING 21 PER CENT.

Wire Diameter, 0.109 in.

External Load, psi.	Cracking Time, sec.	
Grain size, mm....	0.140	0.040
1 070	13.5	135
5 330	7.1	....
10 800	8.6	43.1
11 900	6.0	20.9
19 400	4.9	10.3
23 000	4.5	9.9

TABLE VII.—EFFECT OF GRAIN SIZE ON CRACKING TENDENCY IN MERCUROUS NITRATE SOLUTION OF EXTERNALLY LOADED 0.101-IN. BRASS WIRE DRAWN 35 PER CENT AND RELIEF ANNEALED AT 450 F.

External Load, psi.	Cracking Time, sec.			
Temper......	Hard Annealed	Hard Annealed	Relief Annealed	Relief Annealed
Previous grain size..	0.090	0.020	0.090	0.020
5 330	15	44	....	....
12 400	9	15.2	18.3	314
13 600	8.7	21.8	15.8	114.7
22 000	7.0	12.6	15.4	93.7
25 700	6.2	12.4	12.4	55.6
33 400	....	12.4	....	....
39 700	....	....	....	49.4
41 400	....	....	....	42.8
49 900	....	....	....	34.6

Since, as shown in Fig. 9, the relief anneal did not produce the expected resistance to cracking, specimens of 1 B. & S. No. hard 0.109-in. brass wire

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[7] D. K. Crampton, "Internal Stresses and Season Cracking in Brass Tubes," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., p. 233 (1930).

TABLE VIII.—EFFECT OF RELIEF-ANNEALING TEMPERATURE ON CRACKING TENDENCY IN MERCUROUS NITRATE OF BRASS WIRE COLD DRAWN 21 PER CENT FROM 0.040-MM. GRAIN SIZE.

Wire Diameter, 0.109 in. Time of Anneal, 20 min.

External Load, psi.	Cracking Time, sec.								
	Hard Annealed	350 F.	400 F.	450 F.	500 F.	550 F.	600 F.	650 F.	700 F.
1 070	135	20	....	44.9	49.9	1560	2470	....	....
10 800	43.1	21.8	20.7	51.0	51.2	88.5	337	3020	....
11 900	20.9	20.5	26.2	48.5	86.2	74.9	160	....	....
19 400	10.3	12.7	19.9	15.5	58.8	59.0	105	3220	>80 000
23 000	9.9	10.4	14.0	30.8	75.5	41.0	296	1890	>80 000

that had been drawn 21 per cent from 0.040 mm. were given anneals at temperatures ranging from 350 to 700 F. for periods of 20 min. after attaining furnace temperature. This particular temper was chosen as having the greatest tendency to crack of the drawn material. The results are given in Table VIII and illustrated in Fig. 10, the cracking time being again plotted as a function of the external load for the various conditions. In attempting to catch results during an annealing operation between initial relief of stresses and the final stage, scattering results are to be expected; due to this and the lack of check tests, the data do not warrant an attempt to show anything more than a trend. As was to be expected, little change occurred with temperatures under 450 F. As the temperature of relief annealing was raised, however, the tendency to crack decreased, not suddenly, but in proportion to the temperature of the anneal. As the temperature increased, the wires tended to show greater resistance to cracking with low, external loads, approaching the slope of annealed metal. For comparison, a curve for 0.020-mm. annealed wire has been included. Microscopic examination showed that at 700 F. recrystallization had taken place, and these wires did not crack in over 70,000 sec. It is evident, accordingly, that the degree of relief of stresses in a low-temperature anneal is dependent upon the temperature and time of the anneal, and that since such commercial anneals are given at a temperature and time at which little or no softening occurs, the internal stresses are not eliminated but are merely reduced to a figure that can be regarded as safe for material which is to be put into service. If such materials in service are externally stressed, they will still be more liable to fail than if they had been given an anneal that produced complete recrystallization,

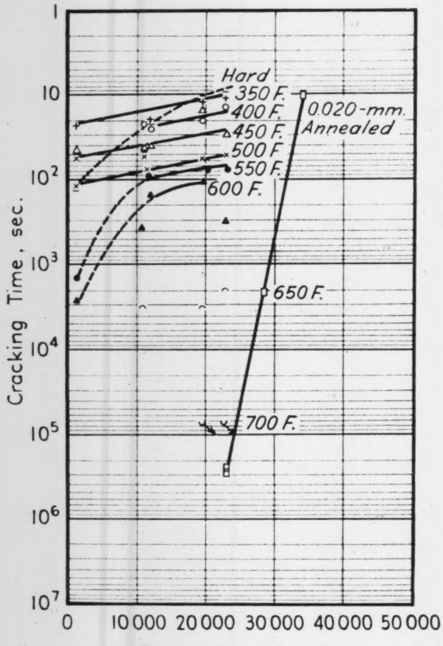

FIG. 10.—Effect of Relief Annealing Temperature on Cracking Tendency in Mercurous Nitrate Solution of Brass Wire Cold Drawn 21 per cent from 0.040-mm. Grain Size.

Wire diameter, 0.109 in.

provided, of course, that the more fully annealed materials were not stressed beyond the yield point.

SUMMARY AND CONCLUSIONS

1. The tendency of brass wire to stress-corrosion crack is influenced by a number of factors exclusive of the surrounding media. The most important are:

(*a*) The magnitude and distribution of the residual stresses.

(*b*) The external loading.

(*c*) The hardness.

(*d*) The grain size.

2. The greater the external load, the greater the tendency to crack. This holds true for annealed, cold-stretched, cold-worked, or cold-worked and relief-annealed wires.

3. The external load increases the tendency of annealed or cold-stretched wires to crack to a considerably greater degree than it does that of cold-drawn wires. The types of these curves are quite different.

4. Wires finished by cold drawing are inherently more susceptible to stress-corrosion cracking than those finished with like reductions by cold-stretching. This holds true up to some point of loading, possibly the yield strength or bearing some relation to that characteristic. This discrepancy between cracking susceptibility of the two types of hardened wires is greater with low external loads.

5. The cracking susceptibility of cold-drawn wires reaches a maximum at a critical temper or degree of cold reduction. The location of this maximum is affected but slightly by the die angle. For the material investigated here, it is in the neighborhood of 20 per cent.

6. The relief of stresses in a low-temperature annealing operation, as indicated by these mercurous nitrate tests on externally stressed pieces, does not take place suddenly and entirely at the point where the hardness and yield strength reach a maximum. On the contrary, these stresses and resulting tendency to crack decrease gradually as the temperature changes from somewhere above room temperature to the point at which complete recrystallization is effected.

7. Grain size not only has a considerable influence on the tendency of annealed wires to fail under loading, but also affects the tendency of drawn wires, cold-stretched wires, and cold-drawn and relief-annealed wires in a like manner and to a considerable degree. In any condition, the larger the grain size, the greater the tendency to fail under corrosion from mercurous nitrate. This influence is so pronounced that quite severe conditions of stress are probably necessary to cause the season cracking in service of brass parts made from small grained metal.

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[See Joint Discussion, p. 927.—ED.]

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JOINT DISCUSSION

MERCURY CRACKING TEST, PROCEDURE AND CONTROL

BY H. ROSENTHAL AND A. L. JAMIESON

INFLUENCE OF EXTERNAL STRESSES ON TENDENCY OF BRASS WIRES TO STRESS-CORROSION CRACK, AS INDICATED BY THE MERCUROUS NITRATE TEST

BY HARRY P. CROFT

MR. HARRY P. CROFT[1] (*presented in written form*).—Messrs. Rosenthal and Jamieson have made a valuable contribution to the knowledge of the mercurous nitrate cracking test by showing the effects of the variables involved.

As mentioned in their discussion of Table VIII, we have obtained results which lead us to believe that the time of cracking in mercurous nitrate is inversely proportional to the temperature of the solution. The range of 50 to 110 F. was investigated, first on drawn cups (the investigation being checked by a second operator), and these results are illustrated by the bottom two curves in the accompanying Fig. 1. Each point is the average of ten or more tests. The cups were drawn from a single strip bar of cartridge brass, the gage and annealing temperature of which had been carefully checked. The first few cups drawn were discarded in order to allow for warming up of the press, so that the parts would be as uniform as possible. The lubricant was removed by an organic solvent, after which the cups were given a 30-sec. sulfuric acid pickle, rinsed three times, and dried. The nature of the draw was such that it resulted in the cups cracking with an audible sound, so that there was very little doubt about the time of cracking. Since these results did not check with the findings at Frankford Arsenal, ten sections from each of two different tubes were tested

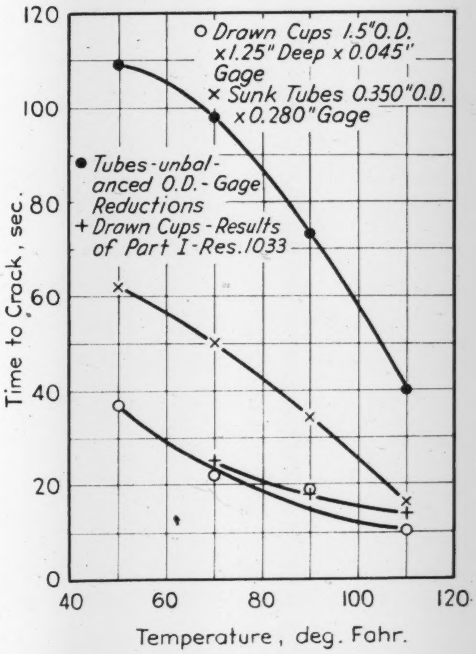

FIG. 1.—Effect of Temperature on Crackability of Stressed 70-30 Brass

at the four different temperatures. These tubes were of cartridge brass, one finished by a sinking operation, the other by a "poor" draw, that is, the diameter reduction was considerably higher than the gage reduction. Six-inch lengths were used, the ends being faced off on a lathe. Samples were entirely immersed. The trend of these curves is all the same, indicating that the higher the temperature, the greater the cracking tendency. Since the action of the solution in promoting cracking is either chemical or electrochemical in its nature, we would expect to find some such temperature influence.

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[1] Assistant Director of Research, Cleveland Plant, Chase Brass and Copper Co., Cleveland, Ohio.

MR. E. H. DIX, JR.[2]—I have been interested in this work largely from the viewpoint of the possible generalization regarding the mechanism of stress corrosion cracking. In the 1940 Institute of Metals Division Lecture, evidence was presented to show that intergranular corrosion is caused by the grain boundaries, either the precipitated phase or the depleted area surrounding it being anodic to the grain areas in the corrosive environment to which the material is being subjected. It would be expected therefore that if this difference in solution potential could be overcome by an applied external potential making the sample the cathode, that stress corrosion could be prevented. The mercurous nitrate test is valuable, it seems to me, as a quick inspection test, but is not particularly desirable from the standpoint of the study of the mechanism of stress corrosion cracking. R. H. Brown, in the Aluminum Research Laboratories, made some measurements of the difference between the solution potential of the grain boundary zones and the grain areas in a 1 per cent ammonium hydroxide solution. He used large-grain samples, which Cyril Stanley Smith, of the American Brass Co., kindly furnished, and a technique previously developed. The large grain size was necessary, so that in one sample the grain boundary zones and in the other the grain areas could be painted out. Brown found a difference in potential of about 0.03 v. in this 70-30 brass, the grain boundary zones being anodic to the grain areas. Also he found a current of around 190 microamperes flowing from the boundary zones to the grain areas.

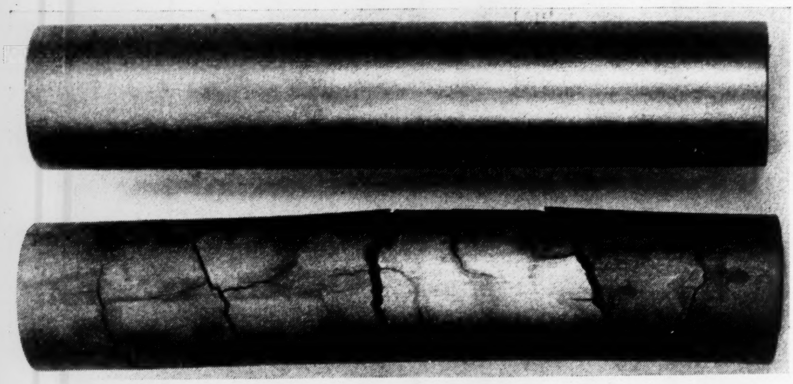

FIG. 2.

There was some interest expressed by the authors of the papers as to whether an applied potential making the sample the cathode would prevent stress corrosion cracking of brass, and Mr. Croft very kindly furnished a 70–30 brass tube which had been sunk so as to have high internal stresses. The tube was $1\frac{1}{4}$ in. in outside diameter and $\frac{7}{8}$ in. in inside diameter and the sample used was about 6 in. long. Using in this case a concentrated ammonia solution, we found that the tube cracked in about 140 hr., as shown in the lower portion of the accompanying Fig. 2. Another piece of the same tubing exposed in the solution but made cathode with an applied potential of 10 v., showed no cracks after 336 hr. as shown in the upper portion of the picture.

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[2] Chief Metallurgist, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

These two experiments would seem to indicate that the stress corrosion cracking or season cracking of 70–30 brass fits into the generalization of the electro-chemical theory briefly stated at the beginning of these remarks.

MR. A. L. JAMIESON[3] (*by letter*).—The data presented in Mr. Croft's paper are extremely interesting and worth while. However, I cannot agree with the author's interpretation of data as presented in his curves.

The author notes in his outline of test methods that some wires elongated (cross-sectional reduction) on loading, but fails to indicate in his data which values were affected by this reaction.

Since the wires most greatly affected by this reduction are the soft wires, it seems that if this reaction were taken into consideration the time to crack of the unstrained (annealed) or slightly strained wires would increase and the curves shown in Fig. 2 of his paper would tend to straighten and flatten out.

The scattering of results in Table III indicates that a number of tests should have been conducted to determine values for the effect of the various degrees of cold work. It might possibly be that had results of sufficient tests been averaged for each point, the average value for each degree of cold work for a given stress would have been of the same magnitude. The curve through such points would have a slope approaching zero which is not in agreement with the author's curves.

The results in Table IV seem to agree with the aforementioned viewpoint. The exceptions of the two highly strained conditions are to be expected due to grain boundary obliteration.

The conclusion from the above interpretation would be that slight strain had very little effect on season or mercury cracking tendencies, thus leaving stress (applied or residual) as the main factor in mercury cracking failures.

The effect of grain size on time to crack seems to be reasonable since small grains would tend to make the path of a crack longer than larger grains and thus increase the time for failure to occur.

MR. CROFT (*author's closure, by letter*).—In regard to Mr. Jamieson's remarks, the results on the wires which elongated visibly during loading were not given special mention since the points for the cracking times fell along the extensions of the curves for the wires which did not stretch, and while this effect was appreciable on some samples, it no doubt existed on others to such a small extent that it was not measurable Also, it was believed desirable to test the samples as rapidly as possible after loading.

The tests on soft wires were not reported in detail as they merely duplicated the work of Morris.[4]

Considerable scattering of results occurred in all this work, and the figures which are given for cracking times represent the averages of from two to ten or more tests.

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[3] Assistant Metallurgist, Ordnance Laboratory, Frankford Arsenal, Philadelphia, Pa.

[4] Alan Morris, "Stress Corrosion of Annealed Brass," *Transactions*, Am. Inst. Mining and Metallurgical Engrs. (I.M.D.), p. 256 (1930).

I appreciate Mr. Jamieson's emphasis of the interpretation in regard to the reason for increasing cracking time with a decrease in grain size.

Mr. Dix's interesting experiments and resulting conclusions seem to indicate the fundamental cause of this phenomenon. Additional potential measurements between the boundary zones and the grain areas, covering metal of the same composition but of different grain sizes, would be helpful in interpreting the results which have been obtained by mercurous nitrate tests.

MESSRS. H. ROSENTHAL[5] and JAMIESON (*authors' closure, by letter*).—The authors wish to thank Mr. Dix and Mr. Croft for their interesting comments. Mr. Dix's data throw considerable light on the mechanism of intercrystalline cracking caused by mercury as compared to that caused by ammonia.

As Mr. Croft points out, increasing the temperature of the mercurous nitrate solution should increase the chemical activity or cracking power of the solution. Supporting this view, Mr. Croft has presented curves following this relationship for tubes and cups. Similar data have been obtained at Frankford Arsenal on time to crack of hard drawn cups tested in a 1 per cent $HgNO_3$ solution at different temperatures.

Stress measurements of caliber .30 cartridge cases, however, have revealed such a complicated stress pattern that it is reasonable to believe small temperature changes of the cartridge case would cause appreciable changes in the stress pattern. This may explain the anomalous behavior of the cartridge cases whose cracking percentage was less at 110 F. than at 70 F.

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[5] Junior Metallurgical Engineer, Ordnance Laboratory, Frankford Arsenal, Philadelphia, Pa.

FINISHES FOR ALUMINUM DIE CASTINGS*

By A. E. Keskulla[1] and Junius D. Edwards[2]

Aluminum die castings are covered with a thin, transparent oxide film which constitutes one of their important advantages from a service standpoint. For numerous applications, the ability of aluminum alloys to form and maintain this natural film is sufficient protection, and further finishing is not necessary. For many other applications, however, where a more decorative appearance or better resistance to service conditions may be desirable, a wide assortment of durable and attractive finishes is available. These finishes are classifiable under the headings:

1. Mechanical finishes, developed by abrading or working the die-cast surface by mechanical means to obtain a polished or textured effect.
2. Chemical finishes, produced by treating the castings with chemical solutions to produce etched surfaces or oxide coatings having a wide variety of properties.
3. Anodic finishes, formed by anodically oxidizing the casting in a suitable electrolyte to form a substantial oxide coating. The oxide coating may then be given supplementary treatments to seal or color it.
4. Electroplated finishes, applied by the electrodeposition of metals such as nickel or chromium on the aluminum surface.
5. Paint and organic finishes, which comprise a multitude of clear and pigmented coatings applied by spraying, brushing, or dipping.

Just which of these methods will be used, or combined, in a commercial finishing procedure for any die casting will depend on such various factors as sales and service requirements, cost considerations, design of casting—size and shape, and alloy choice. Where no background of experience exists, it is advisable for the user of aluminum die castings to consult with the die caster or an experienced jobbing finisher in reaching a decision.

MECHANICAL FINISHES

Mechanical finishing methods are used extensively in preparing die-cast surfaces for other types of finish as, for instance, in the roughing or oiling of castings to remove surface irregularities prior to paint or enamel application, or developing a buffed or textured surface for electroplating or anodic coating. Where mechanical finishes are used without subsequent treatment, they serve a decorative purpose, since they have no substantial effect on abrasion or corrosion resistance. They do not detract from the characteristic resistance of aluminum die castings to ordinary indoor exposures and, in general, they simplify the process of periodic cleaning or polishing.

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* The presentation of this paper at the annual meeting was sponsored by Committee B-6 on Die-Cast Metals and Alloys.

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[2] Assistant Director of Research, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

Polishing:

The sequence of operations generally referred to as polishing may begin with "roughing." This is accomplished with a wheel of glued rag construction, having a working surface of relatively coarse emery particles, 80 to 120 mesh, embedded in glue. Wheel diameters range from 6 to 16 in., thicknesses from $1\frac{1}{2}$ to 3 in. Speeds of about 6000 ft. per min. are suitable for most work. The use of a tallow or composition lubricant will avoid excessive heating and thus prevent breakdown of the glue binder.

"Oiling," sometimes referred to as greasing, is a refinement of the roughing procedure, using finer abrasives and softer wheels. For the average die casting, where flow marks and other surface roughness are not too deep, oiling may be the initial step in polishing. Here the wheels are of solid felt construction, faced with emery of 120 to 200 mesh. Wheel speeds, dimensions, and lubricants are approximately the same as for roughing. Selection of a felt of suitable hardness is important in obtaining the proper work pressure without affecting the contour of the surfaces. Small irregular surfaces require softer wheels than flat, extended sections.

In buffing, wheels are built up from a series of stitched rag buffs, hardness being controlled by the extent and manner of stitching. The abrasive and lubricant are usually in the form of a cake—such as tripoli powder in a grease binder—which is periodically applied to the wheel surface. Pastes containing the abrasive and lubricant are also used, and are applied to the work with a brush. Buffing wheel speeds range from 7000 to 7500 surface feet per minute. Satisfactory results depend upon a proper balance of buff hardness, abrasive compound, wheel speed and work pressure, developed through experience with individual jobs.

"Coloring" produces a surface of high gloss, either as a final finish, or as the foundation for other finishes. Before this operation, it is advisable to remove abrasive from previous steps by means of solvent cleaning, or, where particles have become embedded, by a light caustic etch. Open muslin or flannel wheels are used, to which soft silica or lime is applied as an abrasive. Wheel speeds should equal or slightly exceed those for buffing—7500 to 8000 ft. per min.

Scratch Brushing:

A textured effect of attractive appearance can be achieved by scratch brushing. This may be done on the as-cast surface, following a cleaning operation, or a light acid etch which serves to clean the casting and promote uniformity of finish. Wheels are made with stainless steel, brass or nickel silver wire, about 10 in. in diameter and with wire size from 0.010 in. up, depending on the coarseness of texture sought. Wheel speed should be about 2000 rpm. The removal of metallic particles by a soft brick and periodic reversal of the direction of rotation are necessary to maintain the cutting action of the wires. An acid dip after scratch brushing is frequently employed to remove imbedded metal particles coming from the brush.

Satin Finishing:

Scratch brushing practice may be modified, using smaller softer wheels (0.002 to 0.005 in. wire) to produce a satin finish. The results differ from the scratch brushed surface by having finer lines of less depth and may therefore require an initial oiling treatment to work out surface roughness.

Wheel speeds for satin finishing range from 450 to 600 rpm.

A similar effect is obtainable in the regular polishing routine by substituting for the buffing operation one involving the use of a greaseless satin finishing compound. In this case, the action of the abrasive is to produce fine parallel lines similar to those produced by fine wire brushing.

Ball Burnishing:

Ball burnishing is well adapted to the low-cost handling of parts in quantity, and is a popular method of finishing aluminum die castings. The castings are tumbled, in batch lots, in rotating wood-lined barrels. As a result of the peening action of the steel balls, stars, or other variously shaped members comprising the abrasive charge in the barrels, a bright finish is produced. Barrel speeds of 15 to 35 rpm. and burnishing times ranging from 30 to 60 min. are employed. Ordinarily, shapes and castings in the volume ratio of 2:1 fill the barrel about one half full. Clean water, to which is added a few ounces of neutral soap flakes, brings the level to about two thirds full. It is important that parts be cleaned before burnishing or given a light acid etch followed by a water rinse. To avoid stains, the barrel must be kept free of dirt, grease, heavy metals and their salts, and the solutions should be neutral or very slightly acid. Following burnishing, the castings should be rinsed in clean, hot water and dried in sawdust.

Sand Blasting:

Sand blasting is a rapid method for giving a uniform matte finish to the surface. The texture and color of the sand-blasted surface are determined by the grade of sand or abrasive employed, the rate of introduction of the sand, the air pressure, and nozzle to work distance and angle. The conditions selected must be strictly adhered to if a uniform surface is to be obtained from casting to casting. The use of fine sand (dust blasting) is sometimes employed, particularly as surface preparation for castings which are to be anodically coated. Parts which are to be anodically coated may also require a short treatment with hot caustic soda solution after blasting in order to remove embedded abrasive. This etching treatment also results in a lighter color in the finished oxide coating.

Interesting two-tone effects can be secured by combinations of polishing, buffing, and sand blasting.

CHEMICAL FINISHES

Caustic Etch:

Aluminum die castings can be given a frosted, etched surface by treatment in a hot caustic soda solution. The solution should contain about 15 per cent sodium hydroxide and be used at a temperature of 160 to 180 F. The time of etching will depend upon the effect desired and the concentration and temperature of the solution. After etching, the casting is rinsed in clear, cold water and then given a neutralizing and brightening treatment by immersion in a strong mixture of nitric and hydrofluoric acids. This is composed of eight parts of concentrated nitric acid and one part of concentrated hydrofluoric acid; it should be handled with great care. The presence of the hydrofluoric acid is essential if the alloy contains silicon in substantial amounts. After this treatment the article is again rinsed in clear, cold water and dried on a steam table. The hydrofluoric-nitric acid mixture should be used cold; if it becomes too warm during use it produces a yellow film which is difficult to remove. This acid mixture can be held in a tank lined with carbon brick. Lead is also used, but cannot be considered as permanent.

Alrok Coatings:

Aluminum die castings can be oxide coated by simple chemical treatments, known as Alrok[3] processes. One of these, which is commonly employed, comprises treatment of the die casting in a hot solution of sodium carbonate and dichromate of a predetermined ratio. A substantial oxide coating forms after an immersion period of about 15 min.

This oxide coating gives increased abrasion resistance to the surface, but is much less effective in this respect than a good anodic coating. The protection it gives in corrosive environments can be further amplified by sealing in a hot dichromate solution. In cases where die castings are to be subjected to severe service conditions, it is frequently found desirable to apply such a chromate sealed oxide coating preliminary to painting or enameling the casting.

ANODIC OXIDE COATINGS

Anodic oxidation methods provide means for giving aluminum die castings an oxide coating of substantial thickness and abrasion resistance. Some of these oxide coatings may be used for their decorative effect without further treatment, or they may be given a wide range of colors by impregnation with dyes or mineral pigments. Furthermore, some anodic coatings offer important protection against corrosive environments. As commercially applied, these coatings are usually in the range of 0.0001 to 0.0005 in. in thickness.

The method of application is simple and can be closely controlled. The article to be coated is given the necessary preliminary treatment to clean the surface and give it the desired polish or texture; it is then made anode in a suitable electrolyte. The most commonly used electrolytes are solutions of chromic acid and sulfuric acid in various concentrations. The coatings produced in chromic acid electrolyte are generally thin, of a gray-green color, and cannot be satisfactorily applied to castings containing more than about 5 per cent copper. They are frequently used as surface preparation for painting.

Dilute sulfuric acid is employed in the Alumilite process[4] and the time required for coating ranges from about 10 min. to 1 hr. On pure aluminum, the oxide coating thus formed is transparent or substantially colorless, but the color and appearance may be profoundly modified by the alloying elements always present in die castings.

These effects may become apparent in the hardness, porosity, and protective value of the oxide coating as well as in color and appearance. Depending on their electrolytic behavior, alloying constituents may be anodically dissolved during the coating operation, may be oxidized and remain in the coating, or may remain in the coating in the unoxidized state. Silicon, for example, is a constituent which is not appreciably dissolved or oxidized during the Alumilite treatment and remains in the coating and affects its color and appearance. Die castings with a substantial silicon content are usually gray to black in appearance after being anodically oxidized. Iron constituent also tends to discolor the oxide coating and should be kept at a minimum where light-colored, uniform coatings are desired. Copper constituents, such as $CuAl_2$, also important in die castings, have quite the opposite behavior; these constituents are anodically oxidized and dissolved at a more rapid rate than the aluminum matrix. As a result, the coatings formed on an alloy containing substantial amounts of copper tend to be quite porous and have lower abrasion resistance and protective power because of the anodic solution of the copper constituent. Magnesium forms a white or colorless oxide, but alloys containing substantial amounts of undissolved magnesium constituent have a dark appearance when anodically coated. Alloying elements in solid solution have the least effect upon the color of the casting after oxide coating. Heat treatment of the die casting will affect the amount and degree of dispersion of the partially soluble elements, and hence may have an important effect on the color of the oxidized casting. The oxide coating upon a heat-treated casting is usually lighter in color than on a casting not heat treated.

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[3] Alrok is a registered trademark of Aluminum Company of America.

[4] Protected by patents owned by Aluminum Company of America.

Because of the effect of constituent upon the appearance of the coating, any lack of microscopic homogeneity in the surface structure of the casting may result in a nonuniform, mottled appearance of the coating. Anodically coated die castings may also show the flow lines which follow the course of the metal as it entered the mold. These are due to variations in the structure caused by slight variations in the rate of chill on various parts of the surface. Their elimination or their reduction to a negligible level can sometimes be effected by a change in design of the die and, more particularly, by the selection of an alloy better adapted to receive an anodic coating. A fine sand finish tends to make the flow lines less prominent. In fact, the surface preparation employed prior to anodic coating is an important factor in producing the most attractive finishes.

Anodic coatings can be produced which are porous and will absorb dyes and be colored thereby; they can also be impregnated with mineral pigments by precipitation of the pigment within the pores of the coating. In a similar way, a coating can be given added protection, particularly against salt-containing atmospheres, by sealing in a chromate solution, so that the coating retains within its pores substantial amounts of chromate inhibitor. Coatings sealed in this way usually have a green to yellow color. Other sealing methods are available where coloration is objectionable.

In selecting the alloy best adapted for anodic coating, certain generalizations should be kept in mind. An alloy containing 8 per cent magnesium takes an anodic coating which is light colored and attractive in appearance; the coating is also hard and abrasion resistant. A.S.T.M. alloy VIII (2 per cent copper plus 2 per cent nickel)[5] has a good appearance after anodic coating. Where a dark color is desired, or at least is not objectionable, alloys IV or V (5 per cent and 12 per cent silicon, respectively)[5] may be employed. The substantial silicon contents of these alloys give them a dark color after anodic coating. A.S.T.M. alloy XII (7 per cent copper plus 3 per cent silicon) offers a combination of good casting qualities and a fairly light oxide coating, which can be colored in any except the lighter shades, but has relatively low abrasion resistance.

Evaluation of Oxide Coatings:

The tests selected to determine the quality of oxide coatings will be determined by the intended service of the article. Frequently the thickness of the oxide coat is an important criterion, and the measurement of the electrical breakdown voltage of the coating may yield useful information as to this characteristic. The weight of coating per unit area can usually be closely correlated with thickness. Abrasion resistance can be measured by the abrasive air blast method. Salt-spray tests are usually employed for evaluating the protection against corrosion offered by the coating.

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[5] Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 39 T), 1939 Book of A.S.T.M. Standards, Part I, p. 1147.

ELECTROPLATED FINISHES

Electroplated coatings applied to aluminum die castings serve two major purposes: they provide ornamentation and may give increased abrasion resistance. Resistance to corrosion is not usually improved by plating. However, for mild service conditions, adherent, serviceable and attractive finishes may be obtained by plating.

The actual plating procedures for aluminum alloys do not depart greatly from those used with other common materials. It is important, however, that a suitable preparatory treatment be given in order to insure adherence of the plate, and it is mainly in the details of this preparatory treatment that the commercial plating methods for aluminum die castings require special consideration.

Surface Preparation for Plating:

Acid Etching.—A controlled etch has been found effective in promoting the adherence of electroplates to aluminum base metal. The selective action of the reagents used causes certain of the constituents present in the alloys to be dissolved, leaving microscopic pits or cavities in which the plated metal can find anchorage.

For die castings the etching reagent commonly used consists of:

Nitric acid (sp. gr. 1.42)............	3 parts
Hydrofluoric acid (48 to 52 per cent)...	1 part

After polishing and cleaning operations, the parts are immersed in the nitric-hydrofluoric acid solution, at a temperature of 75 to 80 F., for a period of from 15 to 30 sec. They are then carefully rinsed, and are ready for the plating bath.

Important in this procedure is the proper control of immersion time and solution temperature; they should be definitely established by tests and controlled accurately to produce satisfactory results. Constant solution acidity should be maintained by acid additions.

Krome-Alume Process.—The Krome-Alume[6] process has been successfully applied to the commercial plating of aluminum die castings. In this process the article to be plated is first anodically oxidized in oxalic acid electrolyte; alternating current is commonly employed in this step of the process. The oxidation is carried out at voltages increasing from 5 to 50 v. and for a period of about 10 min.

The coating is then treated in a dilute solution of sodium cyanide or hydrofluoric acid. By this treatment the coating is "modified" and dissolved; the surface is thereby put in good condition for plating by the usual procedures.

Alrok Process.—Another preparatory treatment for die castings which are to be plated comprises the oxide coating of the die casting by an Alrok chemical process, followed by treatment of the oxide coated article in a solution of nitric acid. After a final rinse, the castings are ready for plating.

Plating:

Nickel is commonly plated directly on aluminum, and may be buffed directly to an attractive finish or may be used as a foundation for other metal plates, particularly chromium. The nickel-plating solutions employed are those in general use for the application of nickel electroplating on other metals. They should have good throwing power and give a soft, smooth coating which can be readily buffed to a high luster. The usual lime buffing compositions may be used. Heat treatment of nickel-plated aluminum parts gives improved adhesion of the nickel deposit. For moderate outdoor service, a deposit at least 0.001 in. thick is recommended; generally, the thicker the deposit, the greater the resistance to corrosion.

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[6] Krome-Alume Corp., Lockport, N. Y.

While chromium can be applied directly to aluminum from an ordinary chromium plating bath, the usual practice is to apply it over a preliminary nickel plating. Chromium, when plated over buffed nickel, is more attractive than the duller chromium plate applied directly to aluminum. Heavy deposits of hard chromium plate (0.001 to 0.010 in.) are being applied to aluminum die castings to increase their resistance to abrasion. Other metals, such as copper, brass, and silver, can be applied over nickel plates. Colored or oxidized finishes can be produced, such as oxidized copper, bronze or antique silver; these finishes, as in the case of other metals plated in the same way, should be protected with a clear lacquer. A black finish can be applied by the Moly-Black process,[7] employing a proprietary electrolyte containing salts of molybdenum and nickel.

PAINTING OF DIE CASTINGS

Aluminum die castings may be coated with paint or lacquer, either for decoration or for protection against a severely corrosive environment. When decoration is the principal objective, the preparation of the surface is not usually as important a step in satisfactory paint performance as when protective coatings are needed. However, good resistance to impact and handling is often desired, and hence adequate anchoring of the paint to the surface is important. To secure this, solvent cleaning accompanied by light surface roughening may be sufficient.

The different die-casting alloys of aluminum show some differences in their ability to hold paint under continued humid conditions, although in general these differences are relatively small. With proper attention to surface preparation good results should be secured on any alloy.

There are a number of ways in which the surface of aluminum die castings may be prepared for painting. As mentioned, solvent cleaning is perhaps the simplest form of surface treatment, and for many applications this may be sufficient. The most effective method of solvent cleaning is that of solvent vapor degreasing. A light roughening of the surface, as by sanding, improves the wearing qualities of the finish.

A still more effective method of surface preparation is chemical treatment with a dilute aqueous solution of phosphoric acid. These solutions are more effective if organic solvents are added to the phosphoric acid. The solvents aid in grease removal and permit ready action of the phosphoric acid, which apparently forms a thin film of protective aluminum phosphate on the surface. There are a number of proprietary treatments of this type available.

Alrok coatings, which are applied by a chemical treatment, have been found to be particularly effective on aluminum die castings and they provide an excellent base for the application of paint. These coatings are especially recommended where the service conditions are severe, such as continued service in atmospheres of high humidity. The Pylumin[8] process is a chemical treatment useful in the preparation of the surface for painting. Anodic coatings may also be used under these conditions and in some instances may prove even better. Some of the anodic finishes provide corrosion-resistant, adherent impervious oxide films which constitute ideal bases for paint.

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[7] Process of E. I. du Pont de Nemours and Co.

Following the surface treatment, the metal must be thoroughly dried before paint can be applied. The castings should be heated to a temperature above the boiling point of water for about 30 to 60 min. to insure complete removal of water left on the surface from any aqueous treatment. This is particularly important if a baking enamel or japan coating is to be applied. The castings should receive a minimum amount of handling after cleaning or treating.

Aluminum die castings are usually painted by either dipping or spraying. The method of application to be employed will be determined by manufacturing considerations, and the design of the casting. Some shapes do not lend themselves readily to a dipping operation.

In most cases, the use of a special priming paint is not necessary when the castings are to be painted solely for decorative purposes. Even in such cases, however, the primer should be one which inherently shows good adhesion and which also permits good adhesion of top coats. The value of any painting system depends, to a large extent, upon the selection of a satisfactory primer. In addition to showing good adhesion, the primer should have good resistance to moisture penetration to prevent surface reaction and should contain an inhibitive pigment. Zinc chromate has been found to show the best corrosion-inhibitive properties of any of the pigments which have been tested. Primers in which zinc chromate is the major pigment, if not the only pigment, have been found in most instances to give the best results from the standpoint of protection. Primers of this type are usually made with synthetic resin vehicles which improve their resistance to penetration by moisture. Primers containing lead pigments should not be used in priming aluminum die castings as they have been found to promote corrosion in many cases.

Under most conditions, aluminum paint has been found to be a very satisfactory primer as well as finish coat for use on aluminum die castings. This coating possesses high impermeability to moisture and has good adhesion to the metal.

In many cases it will probably be found desirable to employ baked coatings. The vehicle employed for the primer should therefore be one which will withstand baking at a moderate temperature for at least an hour. Properly formulated synthetic resin varnish vehicles of the phenolic or alkyd types have been found to be quite satisfactory for this purpose.

Where pyroxylin lacquer systems are used in finishing the die castings, special primers must be employed, since many types of primers are "lifted" by the powerful solvents used in lacquer formulation.

With the exception of the special lacquer systems, almost any durable paint or enamel may be employed for the finishing coat after the castings are properly primed. If baked finishes are desired, they should be applied over a baked priming coat. Synthetic resin enamels are particularly effective for this use since they can be formulated to give hard, durable finishes quite resistant to moisture penetration. Where the color of aluminum paint is satisfactory, it is especially useful because of its great opacity and high impermeability to moisture.

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[8] Process of Pyrene Manufacturing Co.

Where speed of drying is a controlling factor, lacquer systems are suggested. Durable lacquer enamels may be secured in any desired color. The more durable lacquers contain a certain amount of synthetic resin. As previously pointed out, special primers should be employed with lacquer systems.

Another type of finish frequently employed for finishing aluminum die castings is the black baking japan. A single coat is often sufficient, but where greater durability is desired, multiple coats may be employed. The japan coatings are baked at a relatively high temperature (usually in excess of 400 F.) and form very hard, abrasion-resistant films which are also quite resistant to moisture and solvents. Similar coatings may be produced with heat-reactive phenolic resins in a variety of dark colors.

In some cases it is desirable to preserve the natural color and appearance of the aluminum die castings. One way in which this can be accomplished is by the use of clear lacquers or synthetic resin finishes. Special lacquers containing appreciable amounts of light-resisting resins such as methacrylate resins, or containing cellulose esters such as cellulose acetobutyrate, have been developed and are especially suitable for this use. Clear varnishes made with alkyd, urea formaldehyde resins or combinations thereof form very satisfactory finishes of the baking type. They are quite hard and abrasion resistant and do not turn yellow in service.

CLEANING AND MAINTENANCE

There are many different reasons for desiring to clean die-cast parts. Naturally, the type of cleaning method selected should be determined by the result which it is desired to accomplish.

Surface Renewing Cleaners:

For some purposes it is desired to remove the surface layer of metal in order to obtain a pleasing appearance. One such method is the "caustic etch," described in another section of this paper. Other such surface renewing treatments include immersion in cold, dilute (1 to 10 per cent) hydrofluoric acid solutions, in cold solutions containing 10 per cent sulfuric acid and 1 per cent sodium fluoride, or in hot 10 per cent to 20 per cent phosphoric acid solutions.

Safe Cleaners:

Surface renewing cleaners should not be used on Alrok or Alumilite-coated articles, or as a rule, on complicated built-up structures, since they will destroy these coatings and are difficult to remove from crevices or other inaccessible regions. Cleaners which cause no appreciable undesirable effect on the articles being treated (that is, safe cleaners) should be employed in such cases. Completely safe cleaners, which can be used in a wide range of concentrations or under widely varying conditions without causing appreciable attack on the aluminum articles being treated, should be used wherever they will satisfactorily accomplish the desired purpose. Most liquid or paste waxes, and most organic solvents (other than the chlorinated hydro-carbons) are completely safe cleaners. Soaps or other alkaline detergents are also often completely safe cleaners, provided they contain a sufficient amount of corrosion inhibitor (such as sodium disilicate or sodium chromate).

Where rapid or powerful cleaning action is desired, alkaline detergents which contain only sufficient inhibitor so that they have no action on the aluminum articles in a limited range of concentrations or conditions can be used. In those cases, the safe operating ranges of such cleaners should be determined by preliminary tests and the cleaners then only employed within safe limits. Chlorinated hydrocarbons, such as ethylene dicholoride, trichlorethylene, and carbon tetrachloride, can also be classed as partially safe cleaners and must be used with care if undesirable action is to be avoided.

General:

In many indoor and some outdoor exposures, bare or Alumilite-coated aluminum die castings require little cleaning or other maintenance in order to retain a pleasing appearance. Periodic dusting or washing with a safe soap is all that is required. Periodic applications of liquid wax are also often satisfactory. In more severe exposures, steel wool can be used in applying the wax in order that adhering deposits will be removed.

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FINISHING MAGNESIUM DIE CASTINGS*

BY H. W. SCHMIDT[1]

Magnesium die castings left to mild atmospheric weathering gradually acquire a layer of oxide, hydroxide, or carbonate which protects them from further attack. For most applications, however, the parts should be finished in some manner. The method adopted will depend on the requirements to be met.

Die castings used for interior applications such as business machines, household appliances, etc., are given finishes chosen for their decorative appeal and wearing qualities.

For the various exterior applications, protective finishing becomes more important and decorative considerations become secondary. For normal inland weathering, where protective requirements are not severe, two or three coats of paint are satisfactory. Aircraft use or marine exposure are more critical, however, and demand the best chemical treatment, inhibitive zinc chromate primer, and maximum protection in the finishing coats.

All phases of metal finishing in use on magnesium die castings are discussed, including preliminary mechanical finishing, cleaning, chemical treating and organic finishing.

MECHANICAL FINISHING

Mechanical finishing methods normally are used to prepare die castings for the application of other finishes. Mechanical finishes are not recommended alone. They should be given some protection.

Polishing:

Surface irregularities are best removed by polishing or grinding. This is accomplished on a built-up cloth wheel "setup" with 100–200 grit abrasive. The peripheral speed normally used is 4000 to 6000 surface feet per minute with 4 to 12-in. wheels. The coarseness of grit will depend on the extent of the polishing operation. Advantage is taken of the very fast cutting action of a dry wheel only where much polishing is required.

To obtain a satisfactory surface on most die castings it is only necessary to use a polishing operation known as "oiling or greasing." The same type of polishing wheels is used at the same speeds and with the same abrasives as noted above. Proprietary commercial greases available on the market are used. The selection will depend primarily on the ease of removal from the die casting in a subsequent cleaning operation. The greases are applied to the polishing wheel and act as lubricants causing a finer cut. The lubricant also aids in preventing the wheel from loading up with fine metal particles. Wheels may be cleaned by using lump pumice. The final polish before buffing should be done on a 220 grit wheel using grease or on a loose buff using a greaseless polishing compound such as a Lea grade L or C. The latter type gives exceptionally good results and is recommended for use on all die castings to be buffed or where only minor imperfections are to be removed. By using greaseless polishing compound, 6 to 10-in. sewed or string buffs running at a peripheral speed of 3600 to 5000 feet per minute gave best results. Buffs should not be too closely sewed; ½ to 1-in. circles or loosely sewed pocket-type buffs are satisfactory.

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Before installing polishing equipment for finishing magnesium, consult the manufacturers of these alloys on the necessary safety precautions to be observed.

Buffing:

Die castings requiring a high luster are buffed using tripoli composition on a loosely sewed buff. Hard sewed buffs cause dragging. Best results are obtained by using approximately 74 to 82 count cloth operating at a peripheral speed of 6000 to 8000 ft. per min. Usually 10 to 14-in. buffs are used. The tripoli used should be free cutting to avoid the necessity of high pressure which tends to cause waviness and dragging.

To produce a very high finish on magnesium die castings, a coloring operation is required using canton flannel buffs. These should be operated at a peripheral speed of 8000 to 12,000 ft. per min. using a dry lime composition and light pressure on a 12 to 16-in. buff. Any polishing or buffing directions must be of a general nature, since so much depends on the requirements of an individual job.

Miscellaneous:

Scratch brushing, sandblasting, shotblasting, and other similar methods of mechanical finishing are used only to a limited extent on magnesium die castings.

CLEANING

Before the application of a chemical coating, the surface must be free of dirt, oil, oxides, and other impurities. The surface should be left in such a condition that it can form a continuous union with the chemical coating to be applied. A thoroughly cleaned surface will be completely wetted when dipped in clean water. A break in the water film is a definite indication that foreign matter exists and the surface is not thoroughly clean.

Oxides or dirt remaining on the surface after degreasing should be removed by a chromic acid cleaning solution.

Solvent Cleaning:

Vapor degreasing methods are satisfactory in many cases where large amounts of oil or grease are to be removed. These methods are not recommended where they are preparatory to some of the chemical treatments described later unless the degreasing step is followed by a cleaning in boiling 2 to 5 per cent caustic soda solution for 5 to 10 min. or by treatment in other strong alkaline cleaners.

Alkaline Cleaning:

Cleaners of the strongly alkaline type, such as commonly used on steel, are the most satisfactory. The procedure may be by boiling or by cathodic electrolytic methods.

A satisfactory degreasing solution for magnesium which may be used either as a boiling or as an electrolytic cleaner is made as follows:

Trisodium phosphate	4 oz.
Sodium carbonate	4 oz.
Soap	0.1 oz.
Water	to make 1 gal.

The soap may be replaced with a suitable wetting agent to ensure freer rinsing. This cleaning solution is operated at 90 to 100 C. When used as a soak cleaner some form of agitation should be used if the bath is operated below the boiling point. The time required for cleaning is 5 to 15 min., depending on the nature and amount of oil or grease to be removed.

As an electrolytic cleaner, the solution may be operated below the boiling point without the necessity of agitation as this is afforded by gas evolution on the surface of the work. The magnesium part is made the cathode in the cleaning solution and a direct current of 10 to 20 amp. per sq. ft. of surface is applied. The only advantage in electrolytic cleaning is that the desired oil or grease removal is affected in a shorter time. Usually 1 to 3 min. are required for complete removal.

Acid Cleaning:

Oxides or dirt remaining on the surface after alkaline cleaning may be removed by a chromic acid cleaning solution. Chromic acid does not attack the metal but will dissolve the oxides. The cleaning bath is made up and operated as follows:

Chromic acid (CrO_3)	1.5 lb.
Water	to make 1 gal.

The parts to be cleaned are immersed for 1 to 5 min. in this bath which is operated at 90 to 100 C. If chloride ions are present in the water the bath functions as a chemical treatment depositing an insoluble film of a different type oxide on the surface, which is detrimental to subsequent chemical treatment. Water which has an appreciable chloride content can be used provided a small amount (usually 0.1 per cent) of silver nitrate is added to precipitate the excess chlorides. The silver nitrate is first dissolved in a small amount of water and then added to the chromic acid bath.

CHEMICAL TREATMENTS

Chemical treatments are used on die castings to provide: (1) an increased corrosion resistance, (2) a base for organic coatings, and (3) a decorative effect.

Chrome-Pickle Treatment:

A chrome-pickle treatment is normally given all magnesium die castings to protect the metal during shipment, storage and machining. The treatment produces a passive surface and etches the metal so that good paint adhesion is assured. For normal service conditions, the coating is a satisfactory paint base. The treatment will remove 0.006 to 0.002 in. of metal from the surface and is, therefore, not recommended on machined surfaces where close dimensions must be held.

Either of the following solutions may be used for this treatment:

(*a*)	Sodium dichromate ($Na_3Cr_3O_7 \cdot 2H_2O$)	1.5 lb.
	Concentrated nitric acid (sp. gr. 1.42)	1.5 pt.
	Water	to make 1 gal.
(*b*)	Chromium trioxide (CrO_3)	1 lb.
	Concentrated nitric acid (sp. gr. 1.42)	0.9 pt.
	Water	to make 1 gal.

The temperature of the solution should be maintained at 125 to 135 F. The time of treatment is 10 sec.

After removal from the treating solution, the die castings should be exposed to the air for about 5 sec. before rinsing thoroughly in cold water and then in hot water to facilitate drying.

Properly applied coatings are matte gray to yellow or iridescent in color. Under the microscope a fine pebbled etch is observed. Coatings to be avoided are bright and brassy in color which under the microscope show a relatively smooth surface with an occasional rounded pit.

Depletion of the solution is indicated by the paleness of the color of the coating, shallowness of the etch, or slowness of the action on the metal. The paleness of color should not be confused with that obtained by insufficient exposure to air between removal from the bath and wash in cold water. Control of the bath is best effected by chemical analysis. The chromate content is determined by titration with lead nitrate. The nitric acid is determined by titration with sodium hydroxide. The methods are as follows:

Chromate Determination.—Pipette 5 ml. of the chrome-pickle solution into a 400-ml. beaker containing 300 ml. of water. Titrate with constant stirring with a $Pb(NO_3)_2$ solution (containing 0.1332 g. $Pb(NO_5)_2$ per ml.) until the yellow precipitate of $PbCrO_4$ ceases to form. Toward the end of the titration the precipitate should be allowed to settle and a drop or two of the $Pb(NO_3)_2$ solution added to the clear supernatant liquor where the precipitation of the $PbCrO_4$ may be readily observed.

Should a precipitate form, stir the solution and repeat the settling operation and addition of $Pb(NO_3)_2$ until the precipitate ceases to form. Note the number of milliliters of $Pb(NO_3)_2$. Each milliliter of $Pb(NO_3)_2$ is equivalent to 0.1 lb. $Na_2Cr_2O_7 \cdot 2H_2O$ per gallon or 0.067 lb. of chromic acid (CrO_3) per gallon. The equations involved in the above titrations are as follows:

$$Na_2Cr_2O_7 + 2\ Pb(NO_3)_2 + H_2 = 2\ PbCrO_4 + 2\ HNO_3 + 2\ NaNO_3 \ldots\ldots\ldots (1)$$

$$CrO_3 + Pb\ (NO_3)_2 + H_2 = PbCrO_4 + 2\ HNO_3 \quad (2)$$

Knowing the volume of the treating bath, the amount of dichromate or chromic acid to be added may be readily calculated.

Nitric Acid Determination.—After completion of the chromate titration with lead nitrate, add an excess of 0.5 ml. of $Pb(NO_3)_2$ solution. Filter off the $PbCrO_4$ precipitate through a plug of absorbent cotton or filter paper. Wash the precipitate with distilled water a few times, collecting the washings in the filtrate. Reject the precipitate. Add 2 or 3 drops of methyl orange indicator to the filtrate and titrate with 1 *N* sodium hydroxide solution (NaOH) until the solution turns yellow. The addition of too much methyl orange renders the endpoint less distinct, particularly with solutions which have been used and revivified several times. Note the number of milliliters of 1 *N* NaOH required for the titration. The equation involved in this reaction is as follows:

$$HNO_3 + NaOH = NaNO_3 + H_2O \ldots (3)$$

It should be noted that in Eqs. 1 and 2 nitric acid is liberated. This must be correct in the calculation for nitric acid in the chrome-pickle solution. The method used for the dichromate chrome-pickle (*a*) is as follows:

$$A - B \times 0.403 \times 0.101 = C$$

where A = total 1 *N* NaOH used, in milliliters,
B = total $Pb(NO_3)_2$ used, in milliliters, and
C = pints of nitric acid (sp. gr. 1.42) per gallon of chrome-pickle solution (*a*).

In the case of the chromic acid chrome-pickle (*b*) the calculation of nitric acid is as follows:

$$(X - Y \times 0.806) \times 0.101 = Z$$

where X = total 1 *N* NaOH used, in milliliters,
Y = total $Pb(NO_3)_2$ used, in milliliters, and
Z = pints of nitric acid (sp. gr. 1.42) per gallon of chrome-pickle solution (*b*).

Knowing the volume of the treating bath, the amount of nitric acid to be added can be readily calculated.

Containers for the chrome-pickle solution may be aluminum, ceramic or other material known to be satisfactory.

All precautions normally observed in the handling of corrosive chemicals should be observed here. Rubber aprons and gloves should be worn by operators.

Sealed Chrome-Pickle:

A modification of the chrome-pickle, which will give increased corrosion resistance, consists in boiling the chrome-pickle coating in a dichromate solution. This treatment is known as the sealed chrome-pickle.

The die casting which has been previously chrome-pickled is immersed in a bath containing 1 to 2 lb. of dichromate (sodium, potassium or ammonium) per gallon for 30 min.

The bath is maintained at boiling temperature.

The dichromate solution is controlled by additions of chromic acid to maintain the pH between 4.0 to 4.4.

Hydrofluoric Acid - Dichromate Treatment:

The color of the coating is brown to black. Practically no change in dimension results from the treatment.

After adequate cleaning, the parts are immersed for 5 min. in a solution containing 15 to 20 per cent by weight of hydrofluoric acid. A satisfactory bath may be prepared by diluting one volume of technical grade 48 to 52 per cent hydrofluoric acid with 2 volumes of water. The usual precautions in handling hydrofluoric acid should be observed.

Rinse thoroughly in cold running water. Boil for at least 45 min. in a solution containing 1 to 1.5 lb. of sodium dichromate per gallon. Rinse thoroughly in cold running water followed by a dip in hot water to facilitate drying.

The hydrofluoric acid bath is only slowly depleted in use. The concentration should not be allowed to fall below 10 per cent. The free acid is titrated with NaOH using phenolphthalein indicator.

Pipette a 2-ml. sample of the hydrofluoric acid solution into at least 100 ml. of distilled water. Add a drop or two of phenolphthalein indicator. Titrate with 1 N NaOH solution until a permanent pink color is produced. The number of milliliters of 1 N NaOH required for the titration is approximately equivalent to the percentage of hydrofluoric acid in the bath. The concentration of the bath should be maintained between 10 and 20 per cent.

A pipette for obtaining the bath sample may be made by lining a 3- or 4-ml. glass pipette with paraffin wax and calibrating to a 2-ml. volume. A rubber bulb should be used for drawing the sample into the pipette.

Depletion of the dichromate bath will be slow and will be indicated by a non-uniformity of coating or an increase in the time necessary to produce a coating. A good control can be obtained by maintaining the pH of the solution between 4.5 to 5.5 by additions of chromic acid.

Containers for the hydrofluoric acid solutions should be lead lined. Some types of rubber lining such as Koroseal have been used successfully.

Steel, aluminum, or most of the commonly used tank materials are satisfactory for the dichromate solution. The dichromate tank should be fitted with a loose cover to minimize evaporation.

Hydrofluoric Acid - Alkaline Dichromate Treatment:

The color of the coating produced by this treatment is brown to black. No appreciable dimensional changes result.

After adequate cleaning, the parts are immersed for 5 min. in a 15 to 20 per cent by weight hydrofluoric acid solution. This solution is identical with the hydrofluoric acid solution in the preceding treatment. Wash thoroughly in cold running water.

The control of the hydrofluoric acid solution is the same as previously outlined under the hydrofluoric acid-dichromate treatment.

Boil the parts for 45 min. in the following solution:

Ammonium sulfate $(NH_4)_2SO_4$	4 oz.
Sodium dichromate $Na_2Cr_2O_7 \cdot 2H_2O$	4 oz.
Ammonia (sp. gr. 0.880)	$\frac{1}{3}$ fluid oz.
Water	to make 1 gal.

The alkaline dichromate bath is controlled by the color of the coatings and pH of the solution. Depletion of the bath is indicated by nonuniform or pale coatings, slowness of coating formation, and by an increase in the pH of the bath to about 6.2. To revivify the bath, add a solution containing equal parts by weight of chromic acid (CrO_3) and concentrated sulfuric acid (sp. gr. 1.84) until the pH is decreased to 5.6. The best operating range of the bath is a pH of 5.6 to 6.0. The original solution level should be maintained by additions of water to replace that lost by evaporation.

Wash thoroughly in cold running water. Boil the parts for at least 5 min. in a solution containing 1 oz. of arsenious acid (As_2O_3) per gallon. The arsenious acid bath (As_2O_3) depletes very slowly. Because of the low cost of this bath, no control is advocated. It is recommended that the bath be discarded periodically, depending on the amount of use.

Chrome-Alum Treatment:

This treatment produces a decorative black finish with very little change in dimensions. The coating is a good paint base but is not as protective as the treatments outlined previously. It is not recommended for prolonged outdoor exposure or severe service.

After adequate cleaning, the parts are immersed in a boiling solution of the following composition:

Potassium chrome alum $(K_2Cr_2(SO_4)_4 \cdot 24\ H_2O)$	4 oz.
Sodium dichromate $(Na_2Cr_2O_7 \cdot 2\ H_2O)$	13.3 oz.
Water	to make 1 gal.

The time of treatment to secure a black color may range from 2 to 15 min., according to the freshness of the solution. Upon removal from the bath, the parts should be rinsed thoroughly in cold running water followed by a hot-water rinse to facilitate drying.

Treatment in a depleted solution results in a superficial layer of brown powder which may be removed by wiping or tumbling in sawdust or other suitable material. The solution may be revivified by additions of sulfuric acid, not exceeding $\frac{1}{3}$ fluid oz. per gal., or sufficient to just redissolve a brown precipitate which settles out during treatment. The solution should be boiled to dissolve the precipitate. The solution may be revived in this manner about five times, after which it should be discarded.

This solution is best controlled by additions of sulfuric acid to maintain the pH between 2.5 and 3.5. The pH of a depleted solution is 5.5.

The containers for the treating solution should be either glass or pure aluminum.

General Considerations:

Selection of the proper chemical treatment will depend largely on service conditions. For interior use, the chrome-pickle coating provides a satisfactory paint base. In many cases, however, no chemical treatment may be needed if the subsequent organic finish is compatible with the metal surface. For ordinary exterior service the chrome-pickle treatment is adequate. Here again it is possible to eliminate chemical treatment, provided a proper choice of primer is used. For severe exterior service, maximum protection and paint adhesion is assured by the use of the hydrofluoric acid - dichromate, hydrofluoric acid - alkaline dichromate, or the sealed chrome-pickle treatments. Appropriate organic finishing systems should always be used on magnesium die castings.

PAINTING

In general, the painting of magnesium die castings follows the rather well-established procedure for other metals. As in all metal finishing, the preparation of the surface is important. This would include proper choice of chemical treatment and adequate cleaning prior to painting.

The chemical treatments discussed elsewhere in this paper ensure a satisfactory surface which should be painted immediately after treatment where practicable. If this cannot be done, the surface usually becomes contaminated, and a final clean-up is necessary before painting. The use of vapor degreasing has proven satisfactory in commercial practice. An alternate method employs a solvent wipe or wash. The solvent chosen should be similar to that used as a diluent for the finish to be applied. The use of hot alkaline cleaners is not recommended, since they might impair the chemical coating.

The finishing schedule can employ either air-dry or baked materials; the choice of paint coatings for magnesium die castings depends on the conditions to be encountered in service.

For interior use, organic finishes normally are chosen for their decorative value. These may be high-gloss japan or enamel systems or may consist of the popular novelty finishes such as wrinkle, crackle, crystal, and the like. Both air-dry and baked finishes can be used. One and two-coat systems can be employed depending upon such variables as surface treatment of die castings, choice of finishing materials, and conditions of service.

Ordinary exterior use of magnesium die castings usually requires a protective finishing system. Decorative considerations are secondary. In fact, the usual decorative finishes are not intended for exterior exposure. Normally, three-coat systems should be used, and the finishing system should be chosen for its ability to withstand the conditions of service to be encountered.

Severe exterior exposure will include aircraft applications, marine exposure and similar applications. For this type of service, two coats of inhibitive primers, such as Navy F-27 or Army 14-080 are recommended, followed by two finish coats. The finish coats for this service should possess maximum impermeability.

Baking schedules have an advantage other than speed-up on drying time, increased adhesion, and resistance to abrasion. Intricate castings may be subject to porous areas with the result that entrapped moisture or air may be detrimental to finishes applied over them. Due to the fact that primers are usually thin coats, a baking schedule will force out any gases from porous areas without harming the primer. In the very few cases where a heavy first coat contains "craters" or "pimples" indicative of minute gas pockets below, it is recommended to preheat the castings long enough at the regular baking temperature to drive off any interfering gases. It is good practice then to apply the finish coat while the castings are still warm.

The recent development of infrared baking has been of considerable importance in speeding up the drying time in commercial finishing. Although baking systems are usually adaptable to infrared radiant drying, paint manufacturers have cooperated by modifying their formulations to conform more suitably to the best practices of this newest method of baking.

Air-dry primers are usually applied over a chemically treated surface. A primer has been developed which has given satisfactory performance when applied to bare metal. Finish coats must be compatible with this primer to ensure satisfaction.

CONCLUSIONS

It has been the aim of this paper to discuss all finishing methods in commercial use on magnesium die castings. Working details have been given wherever possible but in some cases it has been necessary to generalize. In the case of organic finishes, details on baking time, temperatures, and materials are not included since such information is best furnished by the manufacturer. The following table is a summary of the information presented:

Service Conditions	Mechanical Finish	Chemical Treatment	Organic Finish	
			Primer	Finish
Interior	Buffed	None	None	1 to 3 coats of clear lacquer
	None	Chrome-pickle	One coat of baked	1 or more coats air-dry, baked or novelty finish
	None	Chrome-pickle	One coat of air-dry	1 or more coats of air-dry enamel or novelty finish
	None	Chrome-pickle	None	1 or more coats of baked japan or novelty finishes
	None	None	One coat of baked	1 or more coats of baked japan or novelty finish
	None	None	One coat of special air-dry	1 or more coats of compatible enamel or novelty finish
Ordinary Exterior	None	Chrome-pickle	One coat of baked	2 or more coats of air-dry or baked enamel
	None	Chrome-pickle	One coat of air-dry	2 or more coats of air-dry enamel
	None	None	One coat of baked	2 or more coats of baked enamel
	None	None	One coat of special air-dry	2 or more coats of air-dry enamel
Severe	None	Hydrofluoric acid-dichromate	Two coats of baked inhibitive	2 or more coats of baked enamel
	None	Hydrofluoric acid-alkaline dichromate		
	None	Sealed chrome-pickle	Two coats of air-dry inhibitive (P-27)	2 coats of air-dry enamel

GENERAL NOTES.—Baking finishes are preferred to air-dry finishes. The recommendations of manufacturers of magnesium die castings should be followed in the choice of specific organic finishing materials for the above.

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THE FINISHING OF ZINC-BASE ALLOY DIE CASTINGS*

By E. A. Anderson[1]

This paper has been prepared as part of the activities of Subcommittee VI on Finishing Properties of Die Castings of Committee B-6 on Die-Cast Metals and Alloys. In it the attempt has been made to present a general picture of the procedures used in finishing zinc-base alloy die castings and the fields of use to which they apply.

With rare exceptions all of the zinc-base alloy die castings manufactured in this country are made in accordance with the provisions of A.S.T.M. Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 - 38 T),[2] sponsored by Committee B-6. While occasional small differences in finishing behavior are reported between the three alloys of this specification, they may to all intents and purposes be considered alike. What is said here about finishing applies equally to the three alloys.

In the use of any metal, consideration of the need for finishing is based on (1) the desire for decorative effects not obtainable in the metal itself, (2) the need for improved wear and abrasion resistance, and (3) the need for protection against special types of corrosive attack. Plated metallic, plated nonmetallic, immersion nonmetallic, and organic finishes may be used for decoration or for protection. For convenience in presenting the methods used with zinc-base alloy die castings, each of the five main fields is considered separately below.

Plated Coatings for Decoration:

Most of the many zinc-base alloy die castings plated in this country each year are finished in chromium. For light service indoors, and in some cases for interior hardware on automobiles, nickel coatings of 0.0003-in. minimum thickness with an outer chromium coating of 0.00001-in. minimum thickness are applied. For heavier duty in outdoor atmospheres, thicker deposits are required for good service. It is customary to build these thicker deposits with a sequence of copper, nickel, and chromium platings. For exterior automotive service, coatings having a minimum total thickness of from 0.00075 to about 0.001 in. are normally required. The Society's Committee B-8 on Electrodeposited Metallic Coatings has under consideration the adoption of specifications covering deposits of this type on zinc-base alloy die castings.

Die castings usually come to the finishing department with all gates and fins removed. The first stage of preparation for plating is to grind or polish only the areas from which the fins and gates were sheared. This is usually carried out with setup wheels of felt, sheepskin or glued buffs carrying a coating of emery set in place with glue. These wheels operate with peripheral speeds of from 5000 to 10,000 ft. per min. The particle size of the emery is selected to suit the degree of polishing required and the last polishing prior to buffing is normally carried out with a greasy lubricant such as tallow on the wheel.

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[2] Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 - 38 T), 1939 Book of A.S.T.M. Standards, Part I, p. 1153.

Subsequent to any necessary local polishing, the entire casting is buffed on sewed cloth buffing wheels using grease-bound tripoli as the abrasive. These wheels revolve at peripheral speeds of about 8500 ft. per min. The hardness of the wheel, varied by the closeness and frequency of stitching, is selected to suit the particular shape being buffed. Some die castings are subjected to a subsequent coloring operating using a soft, dry, white compound but many finishers merely wipe the casting on a dry portion of the buffing wheel to remove excess grease.

While the plating procedures used with zinc-base alloy die castings are comparable in most respects with those used on other basis metals, the cleaning procedure must be selected with the chemical behavior of zinc clearly in mind. Many plants remove all or most of the grease in a preliminary solvent treatment. Degreasing with trichlorethylene is quite popular but other types of preliminary wash are used to some extent. Subsequent to degreasing, it is common practice to subject the castings to treatment in a mildly alkaline solution. In hand operations, where the time intervals are necessarily short, it is common practice to operate the alkaline solutions as electrocleaners. In many plants the current is passed through the solution to the work evolving hydrogen on the surface of the work. In the past year or so cleaners more suitable for operation with the current reversed, making the work the anode, have appeared and are enjoying considerable use. In conveyorized equipment where longer time intervals are involved, some plants operate without the use of current but depend entirely on the ability of the cleaner to saponify and emulsify the grease present on the surface. Many, however, operate the cleaners with current.

While there are many proprietary alkaline cleaners available for use in the cleaning of zinc, the warning should be given to avoid overcleaning. Overcleaning results in serious difficulty with nonadhesion of subsequently applied electrodeposits. This nonadhesion frequently takes the form of blistering of the coating when the primary deposit is copper and is particularly evident when the parts are heated.

Subsequent to the alkaline cleaning operation, it is necessary that adhering solution, soap, etc., be removed by thorough rinsing and by the action of a dilute acid dip. This is usually either hydrochloric acid or sulfuric acid in concentrations of the order of 1 to 3 per cent by volume. The time of acid dipping is controlled carefully since overtreatment in the acid, like overcleaning, can produce a state of poor coating adhesion. A water rinse is always used after the acid dip and the work is not allowed to dry before plating.

Zinc-base alloy die castings cannot be plated directly with nickel from the warm type of nickel solution commonly used in general plating today. For the relatively thin direct nickel coatings for indoor service, nickel solutions containing substantial amounts of sodium sulfate are used. These have been described in detail in numerous publications. Heavy coatings cannot safely be produced in this type of solution because the nickel tends to crack.

Warm nickel solutions may be used in the deposition of heavy decorative coatings provided the zinc surface has first been covered with a protective layer of electrodeposited copper. This is almost always applied from a cyanide type copper solution. Solutions containing Rochelle salts have been very popular and have been described in some detail in the literature. More recently a proprietary cyanide type solution capable of higher current efficiency than the Rochelle salt solution has received considerable attention.

The nickel solutions used in conjunction with copper primary deposits are those employed with other basis metals and need no description here. Proprietary bright nickel baths are very commonly used. Chromium is deposited over the nickel in accordance with the same standard practice used with other metals.

In a limited number of fields, the final electrodeposit is brass, silver, gold or some other metal. Since all of these metals as well as copper have a strong tendency to diffuse into zinc, relatively heavy coatings[3] (of the order of 0.0003 in.) must be applied or a nondiffusing primary coating must be used. Many brass-plated articles are first given a thin deposit of nickel or a somewhat heavier deposit of copper. The silver, brass, gold, etc., plating solutions used are common to the industry and are not specifically formulated for use with zinc.

There are a number of methods involving the use of current by which semi- or nonmetallic decorative coatings can be produced on zinc alloy die castings. So-called black nickel solutions or a proprietary solution of this type containing molybdenum salts are the most widely used. The black deposits produced are of good uniform color and luster with substantial resistance to wear and corrosion. They may be applied over a primary nickel coating or directly on the zinc surface.

A wide variety of colors can be produced electrolytically on copper electrodeposits on zinc-base alloy die castings.[4] There are available a few methods involving immersion replacement by which a finely divided metallic precipitate can be formed on zinc surfaces, but all of these normally require a coat of clear lacquer or wax to give them reasonable durability.

Plating for Wear Resistance:

For some applications where wear resistance superior to that inherent in the alloy itself is required, chromium deposits of the order of 0.0002 in. in thickness are applied on the die-cast surface. The standard type solution is used but in some cases is operated at room temperature.

Organic Finishes for Decoration:

Zinc-base alloy die castings may readily be buffed to a high luster with a color resembling that of chromium. This finish is not permanent, however, but dulls rapidly outdoors and more slowly indoors. For indoor service where exposure conditions are mild and little abrasion or contact with perspiration is involved, buffed surfaces protected by clear finishes of the type normally used to prevent tarnishing of silver are used to some extent. No successful outdoor applications of this type of finish are known.

Most decorative organic finishes are pigmented or dyed to produce a color effect. Many of these finishes are applied over previously electroplated surfaces to produce a color contrast with the bright chromium plate. Most of these finishes are baked or at least force dried to improve adhesion and speed up production. A wide variety of finishes is available for this purpose but it is considered good practice to use the lowest possible baking temperature to prevent possible undesirable effects on the properties of the die-casting alloys and to minimize blistering of the electrodeposit.

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[3] Absorption of copper deposits by zinc as related to alkaline cleaning practice has been studied intensively. See E. A. Anderson and C. E. Reinhard, "Alkaline Cleaning and Copper Blistering," *The Monthly Review*, Am. Electroplaters' Soc., Vol. 27, May, 1940, pp. 175–183.

[4] U. S. Patents 2,059,053 and 2,081,121.

While a few organic finishes have been formulated specifically for application to cleaned but otherwise untreated zinc alloy surfaces, the majority of organic finishes either do not adhere well initially or lose their adhesion rapidly during service. Commercial experience has shown, however, that sand blasting or treatment in phosphate solutions will produce a surface condition permitting good initial adhesion and good retention of adhesion on the part of the majority of the commercial organic finishes. This is particularly true of the baking type finishes which as a class adhere better than the air-drying type. These pretreatments are relatively inexpensive and open up a wide selection of organic finishes for use in the decoration of zinc-base alloy die castings. The finisher normally selects his finish after due consultation with a recognized finish manufacturer and takes into consideration not only the initial appearance of the finished article but the retention of appearance and the performance during service.

Limited application has been made of combinations of zinc-base alloy die castings and plastics in which thermoplastic materials such as cellulose acetate are injection molded over a core of die-cast metal.

Organic Finishes for Protection:

While the corrosion resistance of zinc-base alloy die castings in warm soapy water is good, the surfaces tend to discolor and become somewhat unsightly. A number of baking finishes have been formulated with synthetic resins, such as phenol formaldehyde, urea formaldehyde and certain of the alkyds, which adhere well to properly pretreated zinc surfaces and are quite resistant to attack by the warm soapy water. Such finishes are used on zinc-base alloy die castings in the washing machine field and permit long service without change in appearance.

Immersion Coatings for Protection:

While zinc-base alloy die castings corrode only slowly under most conditions of contact with moisture, water having little or no access to air attacks them quite rapidly with the production of voluminous corrosion products. In some applications, such as carburetors, these corrosion products are more detrimental than the damage done to the castings by the corrosion. To minimize this type of attack a patented process[5] has been developed which produces on the zinc-base alloy die casting by a simple immersion treatment a golden brown film containing sufficient hexavalent chromium substantially to inhibit attack by stagnant water. This treatment is widely used on carburetors, fuel pumps, and automobile door and trunk locks.

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[5] U. S. Patent 2,035,380.

AN IMPROVED HYDROMETER METHOD FOR USE IN FINENESS DETERMINATIONS

By Alexander Klein[1]

Synopsis

This paper describes a method of test and a hydrometer with a new type of scale which have been developed for measuring the fineness of powdered materials. The hydrometer scale consists of units of surface area rather than the customary units of density. The method of test is generally similar to that usually employed when fineness determinations are made through the use of a hydrometer, but is simplified considerably.

The theory and equations on which the surface-increment scale is based are discussed. Brief methods of computation are illustrated which, while considerably more rapid than those customarily used, yield closely comparable results. Comparisons are made between results obtained by the brief methods of calculation and (1) those obtained by the more detailed hydrometer method used heretofore, and (2) those obtained through the use of the turbidimeter in accordance with the A.S.T.M. Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 – 38 T).[2]

Of available methods for the determination of fineness of subsieve materials, those depending on principles of sedimentation are among the simplest and most convenient for routine relative measurements. Stokes' law, employed in calculating sedimentation diameters, relates the size and velocity of falling particles within fairly well-defined limits. In general, the valid application of Stokes' law is limited to particles of diameter greater than 1 μ.

The applicability of the hydrometer method to analysis of granular materials by sedimentation is well established. However, although the methods generally employed are simple, the operations of preparing the test sample and calculating the fineness are lengthy.

If a sample of material such as portland cement is dispersed in a suitable liquid medium and simultaneous observations of elapsed time and of specific gravity (as measured by a hydrometer) are made as the particles settle, the particle-size distribution and surface area of the sample can be calculated. The method and theoretical considerations have been described in detail in the *Proceedings* of the Society.[3] Briefly, the method heretofore employed consists of the following steps:

1. Observations to determine the specific gravity of a suspension of cement in kerosine, at various sedimentation times.

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[2] 1939 Book of A.S.T.M. Standards, Part II, p. 897.

[3] S. B. Biddle, Jr., and Alexander Klein, "A Hydrometer Method for Determining the Fineness of Portland-Puzzolan Cements," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, pp. 310–331 (1936).

2. For each sedimentation time, computation of
   (*a*) the limiting particle diameter by means of Stokes' law; and
   (*b*) the percentage, by weight, finer than the corresponding diameter.
3. Construction of a particle-size distribution curve from these data.
4. Division of the particle sizes into fractions of sufficiently small range that the mean of the limiting diameters can be taken as the average diameter of the particles.
5. Computation of the surface area of each fraction.
6. Computation of the specific surface (surface area per unit weight of the total sample) by addition of the surface areas of the several fractions.

The simplifications described herein consist of the following:

1. Modification of the hydrometer dimensions to permit more rapid determinations.
2. Substitution of isopropanol (anhydrous isopropyl alcohol, $CH_3 \cdot CHOH \cdot CH_3$) for kerosine as the suspending medium.
3. Substitution of a surface-increment scale for the density scale of the hydrometer.
4. Shortened methods of calculation.

APPARATUS

The apparatus and materials required are as follows:

Surface-scale hydrometer.
Isopropanol.
Uranin (fluorescein sodium salt, technical).
Sedimentation cylinders. Cylinders of proper dimension (46.5 ± 1.0 mm. in diameter and having a volume of 375 ml.) can be obtained by cutting regulation "Kollegiate-Grade" 500-ml. graduated cylinders at the 375-ml. graduation.
Stirring apparatus. The motor and brush specified in A.S.T.M. Method C 115 – 38 T[2] are satisfactory.

With the exception of the hydrometer itself, the apparatus is common laboratory equipment.

Surface-Scale Hydrometer:

The effective depth of immersion of the hydrometer at any sedimentation time, shown as H in Fig. 1, is equal to the depth through which particles of a given size will fall in the given time. The design of the hydrometer is such that its dimensions and the distance H are about one half those of the hydrometers commonly employed; thus the time required for particles to settle the dis-

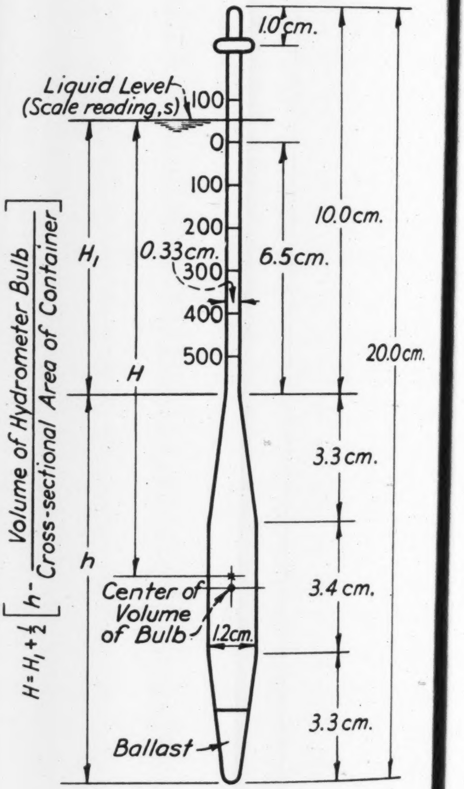
1.0cm.
Liquid Level
(Scale reading, s)
100
0
100
200
300
400
500
0.33cm.
6.5cm.
10.0cm.
20.0cm.
H_1
H
h
3.3cm.
3.4 cm.
3.3 cm.
Center of Volume of Bulb
1.2cm.
Ballast
$H = H_1 + \frac{1}{2}\left[h - \frac{\text{Volume of Hydrometer Bulb}}{\text{Cross-sectional Area of Container}}\right]$

FIG. 1.—Surface-Scale Hydrometer.

tance H is reduced and the total time required for testing is shortened considerably.

Two scales are provided in place of the usual density scale. Surface area is indicated by a surface scale, or "s-scale," which is graduated to 10 sq. cm. The percentage of the total sample finer than a given particle size is indicated by a weight scale, or "W-scale," which is graduated to 1 per cent.

The hydrometer used for portland cement and materials reactive with water is designed for use with isopropanol or liquids of equal density. The dimensions of the hydrometer are given in Fig. 1.

The Liquid Medium:

Isopropanol is of constant composition, and its density and viscosity at any temperature of test need not be determined for each different stock of liquid, but values can be taken from published tables. Materials such as slag, portland cement, and quicklime, which are more or less reactive with water can be dispersed in isopropanol without additional dispersing agents. Materials such as hydrated lime, precipitated chalk, and some paint pigments, which are likely to flocculate in water, are readily dispersed in isopropanol.

To facilitate reading of the hydrometer scales by accentuating the meniscus, a small quantity of uranin is added to the liquid medium at the time of preparing the test sample. For convenience, a 0.5 per cent suspension of the salt in isopropanol can be prepared in advance.

TEST PROCEDURE

For any finely divided material such as cement, a sample for test consists of 14.00 g. The test sample is placed in the hydrometer cylinder together with 200 ml. of isopropanol and 2 ml. of the 0.5 per cent suspension of uranin in isopropanol. The contents of the cylinder are thoroughly mixed by vigorous mechanical stirring for at least 1 min. The volume of the contents of the cylinder is then brought up to the 350-ml. graduation, to produce a final concentration of 40 g. of material per liter.

The sample is then dispersed in the liquid by closing the open end of the cylinder with one hand and alternately inverting and righting the cylinder about 30 times in a period of 1 min. If it appears that all of the material is not dispersed, the process should be continued. As soon as the vessel is righted for the last time, it is placed immediately upon a level table; this instant is taken as the time of start for the test.

A blank sample is made up, similar in all respects to the test sample, but excluding the cement.

At various times after the start of test (sedimentation times), the s-scale of the hydrometer is read. The hydrometer is slowly immersed about $\frac{1}{2}$ min. before each reading is to be taken and slowly withdrawn immediately following each reading, the insertion or withdrawal being made in about 5 sec. Following each observation, the hydrometer is cleansed of any accumulated sediment. Between observations it is advisable to cover the hydrometer cylinder.

The blank sample is observed in conjunction with observations on test samples. When not in use for observations on a test sample, the hydrometer is allowed to remain in the blank sample. The scale reading for the blank sample is determined before each observation of test samples. The temperature of the test sample is determined as soon as the final observation is made.

Five observations are made for each

test sample. The respective sedimentation times t are scheduled so as to correspond approximately to limiting diameters D of 33.5, 19.0, 12.5, 10.0, and 7.5 μ. The sedimentation times used for the five observations, employing the hydrometer of Fig. 1, are approximately 2, 7, 16, 27, and 49 min., respectively.

METHODS OF CALCULATION

For convenience in calculation, the various quantities employed are denoted by symbols, as follows:

Readings on the s-scale of the hydrometer, corrected according to the scale reading for the blank sample, are denoted by the general symbol s, that for the first observation by s_1, that for the second observation by s_2, and so on.

Readings on the W-scale of the hydrometer, corrected according to the scale reading for the blank sample, are denoted by the general symbol W, that for the first observation by W_1, that for the second observation by W_2, and so on.

The diameter of particles corresponding to s and W are denoted by the general symbol D, that measured at the first observation (33.5 μ) by D_1, that measured at the second observation (19.0 μ) by D_2, and so on.

Particle Diameter:

The particle diameter is calculated from Stokes' law from the velocity of fall in a particular settling medium. Since the depth of immersion of the hydrometer depends on the weight distribution as well as on the particle diameter measured at any given time, it is not possible to calculate exactly, in advance of testing, the sedimentation time for a particular size of particle. A close approximation can be made, as described hereinafter under "Theoretical Considerations."

In calibrating the hydrometer, the quantity H is expressed in terms of readings on the s-scale, in the form of an equation such as

$$H = M\,(N - s) \ldots\ldots\ldots (1)$$

in which M and N are constants.

TABLE I.—PARTICLE DIAMETER CORRESPONDING TO SCALE READING s AT TIME t.

Scale Reading, s	Diameter, μ[a]				
	2 min.	6 min., 45 sec.	16 min.	26 min., 30 sec.	49 min.
	1	*2*	*3*	*4*	*5*
0	39.5	21.5	14.0	10.9	8.0
50	38.5	21.1	13.7	10.6	7.8
100	37.8	20.6	13.4	10.4	7.6
150	36.9	20.1	13.0	10.2	7.5
200	35.9	19.6	12.7	9.9	7.3
250	34.9	19.1	12.4	9.6	7.1
300	34.0	18.5	12.0	9.4	6.9
350	33.0	18.0	11.7	9.1	6.7
400	31.9	17.4	11.3	8.8	6.5
450	30.9	16.8	10.9	8.5	6.3
500	29.8	16.2	10.5	8.2	6.0

[a] $D^2 = \dfrac{160\ (1170 - s)}{t, \text{ in sec.}} \ldots\ldots$ (Eq. 3).

The diameter of a particle of material, say portland cement, having a specific gravity of 3.15 and settling in isopropanol can be calculated from Stokes' law by the equation

$$D^2 = \frac{1.837U \cdot 10^6}{3.15 - p} \cdot \frac{H}{t} = K \cdot \frac{H}{t} \ldots (2)$$

where

U = the viscosity,

p = the specific gravity of isopropanol at the temperature of test,

D = the diameter in microns, and

t = the time of settling in seconds.

Equations 1 and 2 can be combined to form the equation

$$D^2 = K\,\frac{M(N - s)}{t} = Q\,\frac{(N - s)}{t} \ldots (3)$$

in which Q is a constant.

For any given value of t, the diameter D can be calculated for any value of s. The values of D thus obtained can be used in the construction of a table such as Table I in which, for each sedimentation time t, values of D are shown corresponding to any s-scale reading.

In Eq. 2 the quantities U and p depend on the temperature of test. For purposes of calculation, the temperature of test is taken as equal to that at which the hydrometer is calibrated. Computed values of D, and hence of the resultant specific surface, require correction if the temperature of test is other than that of calibration.

TABLE II.—SURFACE-AREA-SUMMATION CONSTANTS AND PARTICLE-SIZE DISTRIBUTION OF CEMENTS DISPERSED IN ISOPROPANOL AND TESTED WITH SURFACE-INCREMENT SCALE HYDROMETER.

(Selected values only; complete table should contain about fifty values of s to cover the range properly)

Summation Constants, sq. cm.			Corrected Scale Reading, s	Particle-Size Distribution, per cent Finer than Limiting Diameter				Summation Constant, C, sq. cm.
3-Increment Method, C_3	4-Increment Method, C_4	5-Increment Method, C_5		5-Increment Method, W	4-Increment Method, W_5	3-Increment Method		
						W_4	W_5	
1	2	3	4	5	6	7	8	9
351	352	362	20	4.1	3.1	3.3	2.5	572
522	522	542	50	10.3	7.9	8.6	6.5	1067
810	809	842	100	20.7	16.1	17.3	13.2	1892
1108	1100	1142	150	31.0	24.4	26.2	20.3	2717
1419	1396	1442	200	41.3	32.8	35.2	27.7	3542
1744	1700	1742	250	51.7	41.6	44.6	35.7	4367
2440	2342	2342	350	72.3	60.3	64.2	53.0	6017
3337	3124	2942	450	93.0	83.7	87.4	76.5	7667
3869	3487	3122	480	99.2	95.2	97.8	92.1	8162

Particle-Size Distribution:

The s-scale and the W-scale are proportional to each other. Since it is inconvenient to make observations on both scales at the same time, the values of W corresponding to values of s are tabulated. The arrangement of such a table is illustrated by Table II wherein s-values are given in column 4 and corresponding values of W are given in column 5. For the hydrometer employed in isopropanol suspensions,

$$s = 4.84\, W \dots\dots\dots\dots (4)$$

The s-scale reading for the test sample at any time t is corrected by the amount of the scale reading for the blank sample. If the temperature of test is that of calibration, the scale reading for the blank sample is zero. If the temperature of test is above that of calibration, the scale reading for the blank sample is above zero, and is the amount to be added to the corresponding scale reading for the test sample. If the temperature of test is below that of calibration, the scale reading for the blank sample is below zero and is the amount to be subtracted from the corresponding scale reading for the test sample.

From the corrected scale reading (s-value) at each observation, the corresponding value of W can be obtained from Table II. At the first sedimentation time, s_1 corresponds to W_1 or the percentage finer than 33.5 μ; at the second sedimentation time, s_2 corresponds to W_2 or the percentage finer than 19.0 μ, and so on.

Values of W are not affected by difference between the temperatures of the test sample and that at which the hydrometer is calibrated, provided s-values are used in the calculation of W.

Specific Surface:

From the available particle-size distribution data, the specific surface can be calculated in accordance with established methods. The calculations are simplified through the use of factors related to a surface scale. The first method described below, called the "conventional method," is the most accurate. As compared with the sedimentation time required for the conventional method, the approximate methods, known as the "5-increment," "4-increment," and "3-increment" methods, require sedimentation times in the ratio of 100, 54, and 33 per cent, respectively. The more rapid methods are not necessarily of lower accuracy than the less rapid methods, depending on the particle-size distribution.

Conventional Method.—The calculation of specific surface by the conventional method consists of the following steps:

1. Values of D corresponding to uncorrected scale readings for the test sample are taken from Table I, that for D_1 being found in column 1 opposite the scale reading for the first observation, that for D_2 being taken from column 2 opposite the scale reading for the second observation, and so on.

TABLE III.—CORRECTIONS FOR SPECIFIC GRAVITY AND TEMPERATURE.

Correction for	Uncorrected Value	Coefficient	Corrected Value
Specific gravity, G	W	$\frac{12.15 - G}{9}$	W'
	D	$\frac{7.85 - G}{4.7}$	D'
	S	$\frac{5.25 - G}{2.1}$	S'
	S_1	$\frac{3.15}{G}$	S_2
Temperature of test, T	D or D' S, S', S_1 or S_2	$0.009\ (111 + T_0 - T)$ $0.009\ (111 + T - T_0)$	D_v S_v

2. From column 5 of Table II are taken the corresponding values of W_1, W_2, etc., opposite values of s_1, s_2, etc. in column 4.

3. Values of D are plotted to logarithmic scale against corresponding values of W plotted either to a linear scale or to a logarithmic-probability scale.

4. From the diagram are taken values of W corresponding to particle diameters of 33.5, 19.0, 12.5, 10.0, and 7.5 μ, respectively.

5. The specific surface S is calculated by substituting the values of W taken from the diagram, in the equation

$$S = 4.84\ (W_1 + W_2 + W_3 + W_4 + 6W_5 + 50) \quad \ldots\ldots (5)$$

The calculations described above are for materials considered to have a specific gravity of 3.15 and tested in isopropanol at a temperature T equal to that used in the calibration of the hydrometer (T_0). If the specific gravity G of the material is known, values of W, D, and S computed as described previously are multiplied by appropriate coefficients from Table III to obtain the corrected values W', D', and S', respectively. The specific surface S_1, computed from values of W' and D', is multiplied by the appropriate coefficient to obtain the corrected value S_2. These empirical coefficients are precise when G is equl to 3.15 and are reasonably accurate when G is between 2.4 and 3.7.

When the specific gravity of the material is outside the range of 2.4 to 3.7, or where it is desired to reduce the number of corrections, the original sample weight may be adjusted. For tests in isopropanol, if the concentration is made equal to $\frac{30\,G}{G - 0.79}$ grams per liter, the values of W in column 5 of Table II are applicable. When the adjusted concentration is used, values of D taken from Table I are multiplied by the coefficient $\sqrt{\frac{2.36}{G - 0.79}}$, and computed values of S are multiplied by $\frac{3.15}{G}$.

When the temperature of test is other than that at which the hydrometer is calibrated, values of D and S are multiplied by appropriate coefficients (Table III) to obtain the corrected values D_v and S_v. This method of correction for temperature makes possible the use of the hydrometer of Fig. 1 at temperatures of test between 55 F. and 85 F. The use of the blank sample makes it unnecessary to correct W.

The "5-Increment" Method.—If Eq. 5

be expanded and quantities substituted in accordance with Eq. 4, the combined equation is

$$S = s_1 + s_2 + s_3 + s_4 + 6s_5 + 242 \ldots (6)$$

The quantity ($6s_5 + 242$) can be computed and tabulated for all values of s as a constant C_5, as illustrated in column 3 of Table II. Equation 6 may then be expressed as follows:

$$S = s_1 + s_2 + s_3 + s_4 + C_5 \ldots (7)$$

in which s_1, s_2, etc., are the corrected scale readings (s-values).

The specific surface can be computed directly from the values obtained from the surface scale, without determining the particle-size distribution, and can be corrected for specific gravity and temperature, if necessary, by means of the appropriate coefficients.

The "4-Increment" Method.—It has been established that portions of particle-size distribution curves plotted to logarithmic-probability scales are substantially straight lines, particularly between 5 and 15 μ.[4] The feasibility of limited extrapolation from such a diagram is acknowledged, and values so obtained are considered reasonably accurate. By limiting the number of observations in the hydrometer method to four and estimating the value which would be obtained at the fifth observation, values of specific surface can be obtained which approximate closely those obtained through the use of five observations. Similarly, the number of observations can be limited to three, and extrapolated values can be used for the omitted observations. The method of extrapolation is as follows:

A particle-size distribution curve is plotted, with diameters to logarithmic scale and percentages to logarithmic-probability scale, as a straight line for diameters from 7.5 μ to 12.5 μ, the line having a slope such that the percentage W at 7.5 μ is twice the percentage W at 3.75 μ. A group of such curves is then drawn between limiting curves, where one limiting curve shows the distribution of a material having 99 per cent finer than 7.5 μ, and the other limiting curve shows the distribution of a material having 1 per cent finer than 7.5 μ.

From each curve are determined the ratios

$$R_1 = \frac{W_5}{W_4}, \qquad R_2 = \frac{W_5}{W_3},$$

and

$$R_3 = \frac{W_4}{W_3} \ldots\ldots\ldots\ldots (8)$$

$$\text{Then,} \quad W_5 = W_4R_1 = W_3R_2, \quad \text{and} \quad W_4 = W_3R_3 \ldots (9)$$

Values for each ratio (R_1, R_2, and R_3) are plotted against various values of 4.84 W (or s), those for R_1 being plotted against 4.84 times the corresponding values of W_4 in Eq. 8 and those for R_2 and R_3 being plotted against 4.84 times the corresponding values of W_3.

The last three terms of Eq. 6, that is, $s_4 + 6s_5 + 242$, are rewritten

$$4.84\, W_4 + (4.84)\,(6.0)\, W_4R_1 + 242$$

$$\text{or} \quad 4.84\, W_4\,(1 + 6\, R_1) + 242$$

$$\text{or} \quad s_4\,(1 + 6\,R_1) + 242 = C_4 \ldots (10)$$

Values of C_4 corresponding to any value of s_4 can then be calculated and tabulated as illustrated in column 2 of Table II. Equation 6 can be rewritten

$$S = s_1 + s_2 + s_3 + C_4 \ldots (11)$$

The approximate values of W_5 corresponding to values of s_4 can also be shown, as in column 6 of Table II. It should be noted that s_5, the value at the fifth observation, can be estimated from the value of W_5 by use of the relationship $s_5 = 4.84\ W_5$, as illustrated in columns 4 and 5 of Table II.

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[4] J. B. Austin, "Methods of Representing Distribution of Particle Size," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 11, June 15, 1939, pp. 334–339.

The sedimentation time required for the observation corresponding to a diameter of 10.0 μ (the fourth observation) is only 54 per cent of that required for an observation at 7.5 μ; about 20 min. is saved by omitting the fifth observation.

The "3-Increment" Method.—In a manner similar to that employed in the 4-increment method, the last four terms of Eq. 6, that is, $s_3 + s_4 + 6s_5 + 242$, can be written

$$4.84\ (W_3 + W_3R_3 + 6\ W_3R_2) + 242$$

$$\text{or} \quad s_3 + s_3\ (R_3 + 6R_2) + 242$$

$$\text{or} \quad s_3 + C_3$$

and Eq. 6 can be written in the form

$$S = s_1 + s_2 + C_3 \ldots (12)$$

All values of C_3 corresponding to values of s_3 can be tabulated, as illustrated in column 1 of Table II. Equation 9 can be employed to estimate values of W_4 and W_5 at corresponding values of s_3, as illustrated in columns 7 and 8, respectively, of Table II.

In the application of Eq. 12, only three observations on the test sample are required, observation is made only to a particle diameter of 12.5 μ and values of W at 10.0 and 7.5 μ are estimated. As before, values of s_4 and s_5 for the omitted observations can be computed from values of W_4 and W_5 through the use of columns 4 and 5 of Table II.

The sedimentation time required for the observation corresponding to a diameter of 12.5 μ is only 33 per cent of that required for observation at 7.5 μ; about 30 min. is saved by omitting the fourth and fifth observations.

Special Methods of Calculation:

In the methods of calculating specific surface from the particle-size distribution data, as used herein, the material has been divided for purposes of computation into six fractions between limiting diameters of 124, 33.5, 19.0, 12.5, 10.0, 7.5, and 0 μ. In common with the principles employed in the A.S.T.M. tentative method employing the turbidimeter (C 115 – 38 T),[2] it has been assumed that the surface mean diameter for the fraction finer than 7.5 μ is equal to 3.75 μ; this is equivalent to assuming a linear distribution below 7.5 μ. The use of six fractions in computing specific surface, as employed above, yields results which for the same cement are in fair agreement with results obtained following the A.S.T.M. procedure.

If the material *finer* than 7.5 μ be divided for purposes of computation into six fractions between limiting diameters of 7.5, 5.0, 4.0, 3.0, 2.0, 1.0, and 0 μ, assuming a linear weight distribution, the computed surface area for the material finer than 7.5 μ will be 1.9875 times that obtained when computing the surface area for a single fraction, 7.5 to 0 μ.

In order to compute the *total* specific surface S_p of the material from eleven fractions, of which six are below 7.5 μ, the quantity 1.9875 is taken as 2.0, and the results of observations are then employed in one of the following equations:

$$S_p = s_1 + s_2 + s_3 + s_4 + 16.5\ s_5 + 242 \quad (13)$$

$$S_p = 4.84\ (W_1 + W_2 + W_3 + W_4 + 16.5\ W_5 + 50) \ldots (14)$$

For the same materials, the values of specific surface as computed by Eq. 13 or Eq. 14, will compare closely with those obtained by the air-permeability method.[5]

The last two terms of Eq. 13 can be combined into a single constant and tabulated, as in column 9 of Table II. Values of S_p can be computed, using either the conventional method or the 5-increment method in accordance with the procedures described above and employing either Eqs. 13 and 14 or Table II. Similarly, through the use of values of R determined for the 4-increment and the 3-increment methods, S_p can be calculated through the appropriate use of Table II.

If the particle-size distribution of the material is known over the complete size range to a minimum diameter of 1 μ, the specific surface can be calculated from the expression,

$$S = \frac{61}{4\,G}(W_1 + W_2 + W_3 + W_4 + 2W_5 + 2W_6 + 2W_7 + 6W_8 + 10W_9 + 52W_{10} + 50) \ldots (15)$$

in which W_6, W_7, W_8, W_9, and W_{10} are the respective percentages finer than 5.0, 4.0, 3.0, 2.0, and 1.0 μ.

THEORETICAL CONSIDERATIONS

There follow (*a*) the development of Eq. 5 which is required for calculations by the methods described above, (*b*) the development of the surface-area and weight-distribution scales of the hydrometer, and (*c*) the determination of the sedimentation time required for each observation.

Equation for Calculation of Specific Surface:

The specific surface of any material for which the particle-size distribution is known can be calculated by means of the expression[3]

$$S = \frac{600}{G}\sum\frac{\Delta W}{d} \ldots\ldots (16)$$

where

S = the specific surface, in square centimeters per gram,

G = the specific gravity of the material,

ΔW = the percentage of the material contained between limiting diameters D_1 and D_2, etc., and

d = the average diameter represented by the mean of the limiting diameters D_1 and D_2, etc., for the particular fraction represented by ΔW.

For the separate fractions, $\Delta W_0 = 100 - W_1$, $\Delta W_1 = W_1 - W_2$, etc., with corresponding limiting diameters of D_0 and D_1, D_1, and D_2, and mean diameters d_1 and d_2, respectively. The surface area represented by each fraction ΔS equals

$$\frac{600}{G}\,\frac{(100 - W_1)}{d_1},\quad \frac{600}{G}\,\frac{(W_1 - W_2)}{d_2}, \text{etc.}$$

If for purposes of computation it is assumed that the material consists of six fractions and that the finest fraction has limiting diameters of 7.5 and 0 μ, Eq. 16 can be expanded, each term multiplied by unity, and written

$$S = \frac{600}{G}\left[\frac{0.5}{0.5}\left(\frac{100 - W_1}{d_1}\right) + \frac{1.5}{1.5}\left(\frac{W_1 - W_2}{d_2}\right) + \frac{2.5}{2.5}\left(\frac{W_2 - W_3}{d_3}\right) + \frac{3.5}{3.5}\left(\frac{W_3 - W_4}{d_4}\right) + \frac{4.5}{4.5}\left(\frac{W_4 - W_5}{d_5}\right) + \frac{10.5}{10.5}\left(\frac{W_5 - 0}{3.75}\right)\right] \ldots\ldots\ldots\ldots (17)$$

If values of d are substituted in Eq. 17 such that (see Table IV)

$$0.5\,d_1 = 1.5\,d_2 = 2.5\,d_3 = 3.5\,d_4 = 4.5\,d_5 = 10.5 \times 3.75 \ldots (18)$$

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[5] R. L. Blaine, "Studies of the Measurement of Specific Surface by Air Permeability," ASTM BULLETIN, No. 108, January, 1941, p. 17.

there can be written the expression

$$S = \frac{15.238}{G}(50 + W_1 + W_2 + W_3 + W_4 + 6W_5) \ldots (19)$$

which is the general expression for the specific surface of a material consisting of particles having diameters less than 124 μ. For a material having a specific gravity of 3.15, Eq. 19 reduces to the form shown in Eq. 5. Equations 5, 13, 14, and 19 are applicable to calculation of specific surface from any particle-size distribution data, and are completely independent of the methods used to determine such data.

Specific surface in terms of surface area per unit of *absolute volume* is receiving increasing attention and favor. Such a quantity is useful for the comparison of the particle-size distribution characteristics of several materials on the basis of the same number of particles and is therefore the most representative measure of relative fineness. If, in Eq. 19, the denominator G is replaced by unity, or any value of specific surface per unit of weight be multiplied by G, the resulting value will be the specific surface per unit of absolute volume.

TABLE IV.—MEAN AND LIMITING DIAMETERS TO YIELD INTEGRAL COEFFICIENTS.

Number of Fraction	Maximum Diameter, μ, D_{n-1}	Minimum Diameter, μ, D_n	Mean Diameter, μ, d_n
1	124	33.5	78.75
2	33.5	19.0	26.25
3	19.0	12.5	15.75
4	12.5	10.0	11.25
5	10.0	7.5	8.75
6	7.5	0.0	3.75

Development of Hydrometer Scales:

Where tests are conducted with a hydrometer equipped with a density scale, the percentage W of the original sample composed of particles of size D or less is computed from the equation

$$W = \frac{10^5}{W_0}\frac{G}{G - p}(r - p) \ldots (20)$$

where r is the specific gravity of the suspension, read from the hydrometer scale. The concentration W_0, the specific gravity G of the material, and the specific gravity p of the liquid medium are known constants at a given temperature of test. Therefore Eq. 20 can be expressed

$$W = Z(r - p) \ldots\ldots (21)$$

in which Z and p are constants. Equation 21 shows that W is proportional to $(r - p)$. A W-scale for the hydrometer is constructed such that its zero occurs when r equals p, and such that 100 units on the scale correspond to $Z(r - p)$ at the original concentration W_0. If any concentration W_0 other than 40 g. per liter is used in testing, the scale values of W can be corrected by the factor $\frac{40}{W_0}$.

If the W-scale is multiplied by 4.84 and if 4.84 W_1 is denoted as s_1, 4.84 W_2 as s_2, and so on, Eq. 5 becomes

$$S = s_1 + s_2 + s_3 + s_4 + 6\,s_5 + 242 \ldots (6)$$

The quantity 4.84 W, or s, is in terms of surface area and represents an increment of the total specific surface. Therefore the modified scale, 4.84 W, is a surface-increment scale with limits of 0 and 484 sq. cm. corresponding to limits of 0 per cent and 100 per cent.

Both the W-scale and the s-scale are extended so that the hydrometer can be employed at temperatures other than that of calibration.

Determination of Sedimentation Times:

For a hydrometer having the dimensions shown in Fig. 1 and for material settling in isopropanol, t can be calculated for any given diameter and for various values of s by use of Eq. 3 as illustrated in Table I. If the sedimentation time t is computed for W equal to 0 per cent ($s = 0$) and W equal to 100 per cent ($s = 484$) for *any* diameter D, then the difference in the extreme values of t from the mean value of t, corresponding to the maximum possible error in timing, is 26.0 per cent; and the maximum percentage of error in any diameter is 11.2 per cent if W is between 0 and 50 per cent, and is 13.7 per cent if W is between 50 and 100 per cent. The error in square centimeters per gram is greatest in the fraction finer than 7.5 μ, since most of the surface area is in that fraction.

The surface area of the fraction finer than 7.5 μ is computed from the expression

$$S = \frac{600}{G} \cdot \frac{W}{0.5\,D} \quad \ldots\ldots (22)$$

in which D is 7.5 μ and W is the percentage finer than D. Any error in timing will result in differences between computed and observed values of both W and D. If, for example, the observation is delayed beyond the correct value of t, the computed values of both W and D will be too small. However, the error in the ratio $W/0.5\,D$ will be less than that of either W or D.

The rate of change in the quantity $\frac{W}{0.5\,D}$ depends on the slope of the given particle-size distribution curve; and for any distribution the change in W depends on the change in either D or t. The slope of any given distribution curve can be determined at D equal to 7.5 μ from columns 7 and 8 of Table II. For example, the slope of the distribution curve at W_5 equal to 27.7 per cent finer than 7.5 μ and W_4 equal to 35.2 per cent finer than 10.0 μ, is $\frac{35.2 - 27.7}{10.0 - 7.5}$ or 3.0 per cent per micron.

With maximum error in timing the maximum change in D is $\frac{11.2 \times 7.5}{100}$ or 0.84 μ when W is between 0 and 50 per cent, and the maximum change in W_5 is 3.0 × 0.84 or 2.5 per cent. Also, the maximum change in $\frac{W}{0.5\,D}$ is

$$\frac{27.7 - 2.5}{0.5\ (7.5 - 0.84)} - \frac{27.7}{0.5 \times 7.5} =$$

$$7.57 - 7.40 = 0.17 \text{ which is } \frac{0.17 \times 100}{7.40}$$

or 2.3 per cent. Similarly the maximum errors at W_5 equal to 13.2, 53.0, and 76.5 per cent are, respectively, 1, 4, and 8 per cent. The greatest error occurs when W_5 lies between 70 and 80 per cent. For the average of all of the distribution curves which can be selected from Table II, the maximum error will be about 4 per cent and the mean error about 2 per cent.

For values of W_5 between 0 and 60 per cent the mean errors due to incorrect timing are below 2 per cent. Since most of the materials likely to be tested will have 0 to 60 per cent finer than 7.5 μ, the timing is standardized for a material having a distribution of 72, 56, 45, 38, and 30 per cent finer than 33.5, 19.0, 12.5, 10.0, and 7.5 μ, respectively. On the basis of this timing, comparisons of numerous results calculated by approximate and conventional methods show a maximum error of 4.4 per cent and an average error of 2.3 per cent. The probable error for all conditions will be somewhat lower.

TEST RESULTS

In Table V are shown values of specific surface as computed by approximate methods and by the conventional method. The materials tested include portland cements, portland-pozzuolana cements, and masonry cements. The average difference between results obtained by the 3-increment or the 4-increment method and those obtained by the 5-increment method is less than 2 per cent. The average difference between results obtained by the 5-increment method and those obtained by the conventional method is about 1 per cent.

TABLE V.—SPECIFIC SURFACE DETERMINED BY VARIOUS METHODS OF CALCULATION.

Material	Specific Surface, sq. cm. per g.			
	3-Increment Method	4-Increment Method	5-Increment Method	Conventional Method
Portland cement				
No. 1	1540	1520	1500	1460
No. 2	1880	1870	1840	1840
No. 3	2220	2220	2230	2240
No. 4	2530	2540	2560	2600
No. 5[a]	1920	1920	1910	1910
No. 6	1840	1840	1840	1820
Portland-pozzuolana cement				
No. 7[b]	2160	2120	2110	2120[c]
No. 8[b]	1960	1950	1950	1950[d]
Masonry cement				
No. 9	2010	2080	2020	1980
No. 10	2190	2150	2140	2100
No. 11	2420	2420	2470	2420

[a] Cement Reference Laboratory standard sample No. 114c.

[b] 80 per cent of portland cement No. 6, 20 per cent of pozzuolana.

[c] Specific surface computed from that of constituents, 2100 sq. cm. per g.

[d] Specific surface computed from that of constituents, 1930 sq. cm. per g.

TABLE VI.—REPRODUCIBILITY OF RESULTS OBTAINED BY 5-INCREMENT METHOD.

Operator	Specific Surface, sq. cm. per g.			
	Cement No. 12	Cement No. 13	Cement No. 14	Cement No. 15
A (2 tests)	1468 (9)	1803 (11)	2163 (19)	2498 (0)
B (2 tests)	1460 (13)	1798 (20)	2150 (13)	2487 (20)
C (4 tests)	1496 (15)	1838 (15)	2215 (13)	2557 (15)
Average	1475 (36)	1813 (40)	2176 (52)	2514 (58)
Deviation, per cent of mean				
Maximum	2.4	2.2	2.4	2.3
Average	1.2	1.1	1.2	1.2

NOTE.—Values in parenthesis are maximum deviations from mean values.

Table VI indicates the degree of reproducibility obtained with the hydrometer method employing the 5-increment method of calculation. The samples include four portland cements of different fineness. Tests were conducted by three operators. The experience of two operators was limited to a single demonstration of the test method; the results of the first tests of these two operators are included in the table. The maximum difference between duplicate determinations is 2.2 per cent, the maximum deviation from the mean of all values by all operators is 2.4 per cent, and the average deviation from the mean of all values by all operators is 1.2 per cent.

TABLE VII.—SPECIFIC SURFACE DETERMINED BY TWO TEST METHODS.

Portland Cement	Specific Surface, sq. cm. per g.		
	By Turbidimeter, A.S.T.M. Method	By Hydrometer, Conventional Method	
		Six Fractions	Eleven Fractions
No. 16	1320	1340	1930
No. 17	1495	1520	2350
No. 18	1670	1710	2590
No. 19[a]	1890	1910	3130
No. 20	2200	2240	3700
No. 21	2410	2450	4130
No. 22	2640	2740	4770
No. 23	3120	3180	5680
No. 24	3440	3440	6360
No. 25	4300	4450	8500

[a] Cement Reference Laboratory standard sample No. 114c.

Table VII compares values of specific surface obtained by the hydrometer and employing two methods of calculation, and by the Wagner turbidimeter employing the A.S.T.M. procedure.[2] Values obtained using six fractions for the total material, and employing basic assumptions in agreement with those of the turbidimeter calculations, are in reasonably close agreement with turbidimeter values. The maximum difference between results obtained by the two methods is 3.8 per cent, the average difference is 1.9 per cent, and the average deviation from the mean of all samples is less than 1 per cent. The ratio of specific surface computed from eleven fractions to that computed from six fractions is 1.44 to 1.91; the finer the material the higher the ratio. As previously explained, the possible limits of this ratio are 1.0 and 2.0.

TABLE VIII.—COMPARISON OF MECHANICAL ANALYSES OF SOILS BY TWO HYDROMETER METHODS.

Particle Diameter, μ	Particle-Size Distribution, cumulative per cent finer than indicated particle diameter					
	Soil A		Soil B		Soil C	
	A.S.T.M. Hydrometer[a]	Surface-Scale Hydrometer	A.S.T.M. Hydrometer[a]	Surface-Scale Hydrometer	A.S.T.M. Hydrometer[a]	Surface-Scale Hydrometer
30.0	23.8	24.4	54.3	56.1	66.8	66.1
20.0	21.2	21.2	47.3	48.7	58.1	60.2
10.0	16.8	16.8	36.1	37.3	47.5	47.7
7.5	14.9	14.9	31.8	33.0	43.2	42.8
5.0	12.8	12.4	26.0	26.6	37.6	36.7
3.0	10.4	10.3	19.2	19.9	31.5	30.5
1.5	8.2	7.9	12.6	13.2	25.0	23.2

[a] Standard Method of Mechanical Analysis of Soils (D 422 - 39), 1939 Book of A.S.T.M. Standards, Part II, p. 453.

Table VIII is a comparison of the particle-size distributions of three soil materials. With the A.S.T.M. hydrometer the standard procedure was used. With the surface-scale hydrometer, the testing procedure was substantially the same, but the specified water-bath temperature control was not used, and calculations were made in accordance with the methods described herein. In no case do corresponding values differ by more than 2.1 per cent of the total sample.

Table IX indicates the wide applicability of the hydrometer method. Included are results for certain materials of abnormal particle-size distribution for which relatively large differences exist between values obtained by the 5-increment method and those obtained by the

TABLE IX.—SPECIFIC SURFACE OF MATERIALS OF VARIOUS TYPES.

Material	Specific Gravity	Liquid Medium	Specific Surface				
			Weight Basis, sq. cm. per g.				Volume Basis, sq. cm. per cu. cm.
			5-Increment Method	Conventional Method			Conventional Method
			Six Fractions[a]	Six Fractions[a]	Eleven Fractions		Eleven Fractions[a]
					Assumed[a]	Observed[b]	
Fly ash	2.54	Water	3200	3250	5 670	6 000	14 400
Pumicite	2.35	Water	3070	2910	3 500	3 920	8 200
Silica	2.65	Water	2850	2870	4 830	4 590	12 800
Clay	2.65	Water	3020	3060	5 690	11 000	15 100
Raw mix A (dry process)	2.72	Water	3240	3250	5 930	6 260	16 100
Raw mix B (slurry)	2.70	Water	3050	2970	5 490	7 345	14 800
Slag A	2.90	Isopropanol	3720	3780	5 840	6 260	16 900
Slag B	2.91	Water	2780	2990	5 290	6 030	15 400
Calcium carbonate							
Coarse fraction	2.75	Water	1560	1620	1 960	1 520	5 400
Medium fraction	2.75	Water	3640	4010	6 930	....	19 000
Fine fraction	2.82	Water	4450	4380	8 490	8 740	23 900
Dicalcium phosphate							
Coarse fraction	2.36	Water	1660	1605	1 860	1 480	4 400
Fine fraction	2.36	Water	5180	4950	9 860	9 700	23 300
Tricalcium phosphate	2.68	Water	3840	4000	6 900	7 700	18 500
Aluminum powder	2.50	Isopropanol	5200	5550	10 400	....	26 200

[a] Assuming linear distribution below 7.5 μ.
[b] Based on observations to 1 μ; see Eq. 15.

conventional method. In this connection, attention is called to the condition that values of *specific surface* as determined by different types of apparatus may agree closely, yet the *particle-size distribution* can vary considerably.

In the last column of Table IX are given values of specific surface on a basis of surface area per unit of absolute volume. Since particle size as between different materials is essentially a function of volume rather than weight, the volume basis is more useful than the weight basis for comparative purposes.

CONCLUDING REMARKS

It is recognized that the limitations in the application of the hydrometer to the determination of fineness are in accordance with the limitations in the use of Stokes' law; to this extent the usefulness of any sedimentation method is limited. There are several inherent advantages in the hydrometer method which justify its use in both practical and theoretical applications. The principal advantages of the hydrometer and the hydrometer method are:

1. The results obtained by hydrometer methods are substantially independent of all properties of a material other than those properties directly related to its fineness, namely, size, shape, and distribution.
2. No mechanical or electrical equipment (other than the stirring apparatus) is required.
3. The characteristics of the hydrometer do not change, and only an initial calibration is required.
4. Particle-size distribution can be obtained by hydrometer methods with a speed and accuracy which are the equal of any other present method.

The method described herein conforms in all essential respects to the principles and applications developed by previous investigators including Boyoucos, Casagrande, Hogentogler, Willis and Johnston, and others. The conventional method of calculation is a highly condensed form of a method of established standing. The dependability of the apparatus and method are independent of the assumptions made with respect to the surface mean diameter of the finest fraction; these assumptions have been made by the author merely in order that values of specific surface as determined by the hydrometer shall be of the same order as those determined with several other types of apparatus. There are also provided methods of calculating specific surface which yield values believed by some investigators to approach more closely to the true values, and methods of calculating the specific surface in units of absolute volume. The approximate methods of calculation have been included in the interest of a wider and more economical application of the hydrometer. The great bulk of commercially produced powders have normal particle-size distributions, and the use of the approximate methods for the determination of fineness of such materials is justifiable and desirable.

Acknowledgments:

Acknowledgments for valuable services and active cooperation in connection with the development of the apparatus and method of test described herein are due Samuel Schultz and C. E. Walcott, senior students in Civil Engineering at the University of California. J. W. Kelly, Lecturer in Civil Engineering, cooperated in the preparation of the report. Special acknowledgment is due Raymond E. Davis, Director of the Engineering Materials Laboratory, for constructive guidance.

EFFECT OF FREEZING TEMPERATURE IN FREEZING-AND-THAWING TESTS OF BRICK

By Joseph C. Richmond[1] and John W. McBurney[1]

Synopsis

This paper reports the effect on the resulting disintegration of two samples of half bricks, of freezing and thawing for 50 cycles using freezing temperatures of 20 F. and −20 F., respectively. These two samples were formed by breaking bricks of a single sample into half bricks. The sample included a picked group of bricks formed by the dry-press, soft-mud and stiff-mud processes and was similar to samples tested in 1938.[2]

The effect of certain variations in the method of making freezing-and-thawing tests on the resultant disintegration of selected and presumably similar samples of brick was reported in 1938.[2] The influence of intensity and speed of freezing had not been investigated for the reason that the equipment then available did not provide for temperature control. Subsequent to the publication of these results[2] the National Bureau of Standards built a freezing apparatus conforming in principle to that described by Scholer and Stoddard[3] and meeting the requirements of the apparatus described in the Society's Tentative Method of Test for Soundness of Aggregates by Freezing and Thawing (C 137 - 38 T).[4] This equipment provided a constancy and reproducibility of freezing conditions and temperatures unobtainable in the ordinary freezing chamber and thus made possible the tests reported in this paper.

Preparation and Properties of Samples of Brick

The 1938 paper[2] described in detail the preparation and properties of a series of twelve samples of bricks and reported the results of freezing and thawing on eleven of these samples. In summary, samples 1 to 7 were frozen and thawed in the laboratory by methods 1 to 7; samples 8 to 10 were subjected to weathering by outdoor exposure and sample 11 was used as a check against sample 1 since it was frozen and thawed by the same method (method 1). In the present investigation bricks from the remaining sample (12) were broken transversely and the two sets of halves constitute the current samples *a* and *b*. Sample *a* was frozen by method *a* (20 F.) and sample *b* by method *b* (−20 F.).

Five types of brick are included, as follows: Brick A is a salmon dry-press

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[1] Junior Technologist, and Senior Technologist, respectively, National Bureau of Standards, Washington, D. C.

[2] J. W. McBurney and A. R. Eberle, "Freezing-and-Thawing Tests for Building Brick," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 470 (1938).

[3] C. H. Scholer and A. E. Stoddard, "Proposed Method of Testing Concrete and Concrete Aggregates by Freezing and Thawing," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 364 (1932).

[4] 1939 Book of A.S.T.M. Standards, Part II, p. 990.

TABLE I.—COMPARISON OF EFFECTS OF FREEZING AT 20 F. AND AT −20 F. ON A SAMPLE OF A DRY-PRESS CLAY BRICK (BRICK A).

Specimen Number of Whole Bricks	Saturation Coefficient of Half Bricks		Loss in Weight as Percentage of Original Dry Weight, or Number of Cycles to Breakage (B−c)					
			25 cycles		34 cycles		50 cycles	
	Sample *a*	Sample *b*	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.
1A......	0.817	0.820	0.17	0.18	0.26	0.27	0.68	0.45
2A......	0.816	0.822	1.29	0.94	1.57	1.13	2.12	1.60
3A......	0.842	0.838	1.22	0.65	1.39	0.74	2.35	1.20
4A......	0.851	0.839	0.55	B21c	0.64	....	1.11	....
5A......	0.843	0.843	0.37	B22c	0.56	....	0.65	....
6A......	0.846	0.835	0.75	1.52	1.00	B30c	1.33	....
7A......	0.844	0.843	0.59	1.01	0.76	B32c	0.93	....
8A......	0.844	0.839	0.64	0.76	0.79	0.98	1.03	B35c
9A......	0.852	0.843	0.87	0.76	1.09	1.10	1.53	B36c
10A......	0.859	0.854	0.50	0.85	0.80	B33c	1.09	....
11A......	0.855	0.844	2.98	1.64	3.47	B32c	3.87	....
12A......	0.848	0.842	0.26	0.57	0.52	0.85	B46c	B37c
13A......	0.853	0.856	0.44	B22c	0.61	....	0.87	....
14A......	0.845	0.837	0.29	0.25	0.49	0.25	0.68	B38c
15A......	0.852	0.853	0.58	0.34	0.78	0.52	0.97	B35c
16A......	0.863	0.854	0.83	0.78	1.47	B29c	1.74	....
17A......	0.867	0.861	0.76	1.84	0.93	B32c	1.34	....
18A......	0.869	0.855	0.94	1.41	1.17	1.62	1.84	B40c
19A......	0.857	0.860	0.97	0.56	1.65	B29c	2.08	....
20A......	0.878	0.874	0.48	0.48	0.61	1.06	1.03	B40c
Avg. . .	0.850	0.846						

TABLE II.—COMPARISON OF EFFECT OF FREEZING AND THAWING AT 20 F. AND −20 F. ON A SAMPLE OF A SIDE-CUT SHALE BRICK (BRICK B).

Specimen Number of Whole Bricks	Saturation Coefficient of Whole Bricks[a]	Loss in Weight as Percentage of Original Dry Weight, or Number of Cycles to Failure (F−c) or Breakage (B−c)					
		25 cycles		34 cycles		50 cycles	
		Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.
1B	0.800	1.00	0.96	1.36	1.24	1.79	1.92
2B	0.805	1.59	2.05	2.72	3.30	3.78	4.54
3B	0.810	1.15	2.18	1.86	3.16	2.57	4.59
4B	0.815	3.38	1.43	9.01	1.83	F34c	2.79
5B	0.815	F21c	F6c	....	....	....	....
6B	0.820	F17c	F8c	....	....	....	....
7B	0.815	F24c	F11c	....	....	....	....
8B	0.825	1.04	1.78	1.49	2.18	1.87	B43c
9B	0.825	1.67	4.29	3.82	8.31	B36c	F35c
10B	0.830	1.27	1.60	1.63	1.91	6.88	B48c
11B	0.825	F9c	F20c	....	....	....	....
12B	0.835	4.03	F17c	F32c	....	....	....
13B	0.835	5.67	F6c	F27c	....	....	....
14B	0.845	F2c	F2c	....	....	....	....
15B	0.840	F11c	F7c	....	....	....	....
16B	0.845	F17c	F10c	....	....	....	....
17B	0.845	F22c	F14c	....	....	....	....
18B	0.840	F2c	F4c	....	....	....	....
19B	0.850	F10c	F4c	....	....	....	....
20B	0.855	F9c	F2c	....	....	....	....

[a] Since these specimens showed evidence of disintegration by the 5-hr. boiling test, saturation coefficients were not determined on the half bricks. The values given are those determined in 1938[2] and have been rounded to the nearest 0.005.

TABLE III.—COMPARISON OF EFFECT OF FREEZING AND THAWING AT 20 F. AND −20 F. ON A SAMPLE OF A SOFT-MUD CLAY BRICK (BRICK C).

Specimen Number of Whole Bricks	Saturation Coefficient of Half Bricks		Loss in Weight as Percentage of Original Dry Weight, or Number of Cycles to Failure (F−c)					
			25 cycles		34 cycles		50 cycles	
	Sample *a*	Sample *b*	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.
1C......	0.637	0.650	0.22	0.36	0.37	0.72	0.74	1.07
2C......	0.645	0.647	0.67	0.48	1.01	0.67	1.34	0.87
3C......	0.670	0.675	0.39	0.50	0.49	0.67	0.98	0.83
4C......	0.651	0.676	0.83	0.46	1.19	0.61	1.43	0.91
5C......	0.694	0.699	0.46	0.30	0.68	0.52	1.03	0.97
6C......	0.701	0.709	0.28	0.53	0.36	0.79	0.64	1.32
7C......	0.726	0.737	0.31	0.32	0.39	0.76	0.86	1.08
8C......	0.738	0.738	0.79	0.34	0.99	0.43	1.58	0.77
9C......	0.745	0.757	0.33	0.32	0.50	0.32	1.00	0.74
10C......	0.715	0.750	0.52	0.56	0.65	0.64	1.17	1.20
11C......	0.763	0.766	F6c	F7c	....	....	....	....
12C......	0.763	0.750	0.34	0.72	0.42	0.93	0.76	1.78
13C......	0.773	0.769	F17c	5.99	....	F34c	....	....
14C......	0.765	0.760	4.25	2.52	6.07	4.11	7.19	7.47
15C......	0.796	0.789	F1c	F2c	....	....	....	....
16C......	0.766	0.812	2.27	3.06	3.57	4.60	4.79	8.58
17C......	0.782	0.789	F3c	F2c	....	....	....	....
18C......	0.788	0.810	F1c	F1c	....	....	....	....
19C......	0.796	0.804	F2c	F2c	....	....	....	....
20C......	0.816	0.815	F3c	F2c	....	....	....	....
Avg. . .	0.736	0.745						

TABLE IV.—COMPARISON OF EFFECT OF FREEZING AT 20 F. AND AT −20 F. ON A SAMPLE OF A SIDE-CUT CLAY BRICK (BRICK D).

Specimen Number of Whole Brick	Saturation Coefficient of Half Bricks		Loss in Weight as Percentage of Original Dry Weight, or Number of Cycles to Failure (F−c) or Breakage (B−c)					
			25 cycles		34 cycles		50 cycles	
	Sample *a*	Sample *b*	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.
1D	0.853	0.847	0.30	B13c	0.30	....	F48c	....
2D	0.832	0.855	0.24	B11c	0.24	....	0.39	....
3D	0.864	0.853	0.41	B19c	0.41	....	0.74	....
4D	0.868	0.864	0.72	B15c	0.72	....	0.72	....
5D	0.884	0.875	0.60	B17c	0.60	....	B46c	....
6D	0.874	0.867	B15c	B10c	....	....	....	....
7D	0.869	0.878	0.50	B14c	0.50	....	0.60	....
8D	0.869	0.883	0.28	B12c	0.28	....	0.56	....
9D	0.899	0.900	0.35	B11c	0.35	....	5.13	....
10D	0.873	0.896	4.36	B5c	F34c	....	....	....
11D	0.885	0.891	0.30	B13c	0.30	....	0.50	....
12D	0.895	0.885	0.34	B12c	0.94	....	7.12	....
13D	0.894	0.898	0.40	B7c	0.40	....	0.56	....
14D	0.888	0.885	1.00	B4c	1.09	....	F46c	....
15D	0.896	0.909	0.54	B8c	0.54	....	0.54	....
16D	0.890	0.889	2.20	B8c	2.27	....	F50c	....
17D	0.897	0.890	0.46	B11c	2.76	....	5.44	....
18D	0.892	0.897	F17c	B7c	....	....	....	....
19D	0.899	0.897	5.12	F3c	5.20	....	F50c	....
20D	0.892	0.896	0.23	F1c	0.39	....	F42c	....
Avg. .	0.881	0.883						

clay brick. The manufacturer of brick B, a side-cut shale brick, characterized it as an "unmarketable salmon." A wide range of burning, from very soft salmons to hard, durable brick, is represented by the soft-mud clay brick, brick C. Brick D is a laminated side-cut shale brick. This brick is a salmon, even though it is much stronger than the other bricks included in the sample. Brick E is a salmon end-cut brick which is also laminated.

The saturation coefficients for both sets of half bricks A, C, D, and E were determined and are reported in Tables I, III, IV, and V, respectively. Half bricks B were not tested because the 5-hr. boiling test had a disintegrating effect on them. However, the saturation coefficients for the whole bricks, as determined in the former investigation,[2] are given in Table II.

The five types of brick (A to E) represented in these samples had been further studied by Stull and Johnson and Table I of their paper[5] presents data on the saturation coefficients of these bricks.

METHODS OF TEST

Saturation coefficients were determined by the procedure described in the Society's Standard Methods of Sampling and Testing Brick (Modulus of Rupture, Compressive Strength, Absorption) (C 67 - 39).[6]

Preliminary tests on 2-in. cubes cut from bricks had indicated that exposing samples in trays nested in a sheet-metal container had been relatively ineffective in securing quick freezing when the container was immersed in an alcohol-water mixture as prescribed in Method C 137.[4] Therefore the following procedure was adopted: All half bricks were oven dried, cooled, and weighed to the nearest 1.0 g. They were totally immersed in water at 70 F. for 4 hr., then placed in rubber bags and enough water added so that the bricks, while standing on edge, were partially immersed in from $\frac{1}{2}$ to 1 in. of water. The bags were sealed and immersed in the alcohol-water freezing mixture so that free circulation of the freezing medium took place around each bag. After freezing for 19 hr., the bags were totally immersed in water (initial temperature 70 F.) for 1 hr. after which the bags were opened and immersion continued for an additional 4 hr., during which the bricks were removed from the bags, inspected, and replaced. The bags were then sealed and the cycle of freezing and thawing repeated. After completion of each fifth cycle the specimens remained immersed in water for 48 hr. (Saturday noon to Monday noon).

After completion of 25 cycles, all surviving specimens were dried in an oven at approximately 240 F. for 72 hr. and reweighed. Drying and weighing were repeated after completion of 34 cycles and again after 50 cycles.

Specimens were withdrawn from the test when they failed by breakage or when they were estimated by visual observation to have lost 10 per cent of the original dry weight. Results are expressed as the number of cycles causing failure (F-c) or breakage (B-c), or, for the bricks that survived, as the loss in weight caused by 25, 34, and 50 cycles, expressed as a percentage of the original dry weight.

The rate of freezing on one specimen under various conditions was observed by means of a copper-constantan thermocouple placed into a hole drilled into the center of the specimen. Figure 1 illustrates the time-temperature curves for this specimen sealed in a rubber bag and exposed in the alcohol-water freezing mixture to temperatures of 20 F. and of

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[5] R. T. Stull and Paul V. Johnson, "Saturation Coefficient Values for Brick by the Absorption-Boiling and Absorption-Porosity Methods," ASTM BULLETIN, No. 109, March, 1941, p. 17.

[6] 1939 Book of A.S.T.M. Standards, Part II, p. 104.

−20 F., and when exposed on edge in $\frac{1}{2}$ in. of water to the air of the freezing chamber used in the work reported in 1938.[2]

It will be noticed that the −20 F. curve and that for the brick exposed to the open-air freezing chamber show a secondary hesitation or plateau at about 27 F. This indicates that the freezing point of the water in the center of the brick has been depressed by about 5 F. Calculation shows that a pressure in the neighborhood of 300 atmospheres, or approximately 4500 psi., would be required to depress the freezing point of distilled water to this extent. This pressure is in the same order of magnitude as the compressive strength of the brick in question. The observed depression of the freezing point may be accounted for to a certain extent by dissolved salts, both in the tap water used and from soluble salts in the brick, but in spite of that there is undoubtedly a considerable pressure developed. Thomas,[7] by dilatometer and calorimeter determinations, found that all the water contained within the pores of building stone is not frozen at one temperature, but that in most cases an appreciable quantity may still be unfrozen at −5 C. (23 F.). The explanation given is that as the surface water is frozen the interior water is entrapped and subjected to pressure which lowers the freezing point.

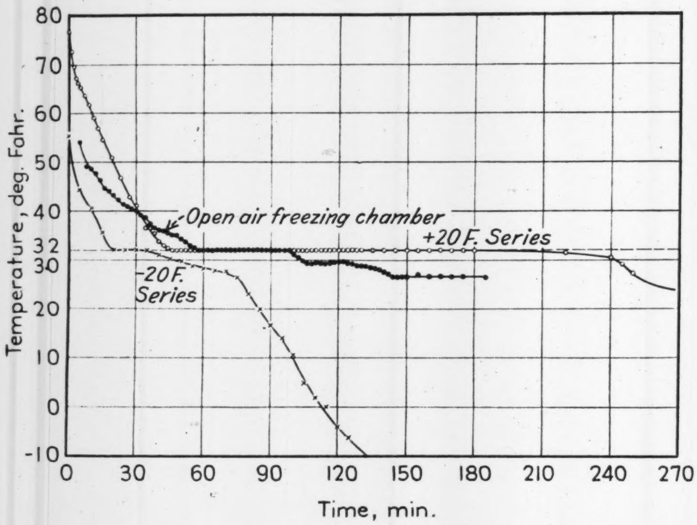

FIG. 1.—Time-Temperature Curves for Various Conditions of Freezing.

DISCUSSION OF DATA

Similarity of Samples.—Since samples 12*a* and 12*b* are composed of the respective half bricks of a picked sample of whole bricks, differences in results of the freezing-and-thawing tests may be ascribed, with considerable confidence, to differences in test methods rather than to differences in specimens. Further assurance as to the similarity of samples *a* and *b* is provided by the agreement between the saturation coefficients of the pairs of half bricks. With the exception of 4C, 10C, 16C, 2D, and 10D, all of the pairs of bricks A, C, and D checked within ±0.01 which is the reproducibility of the saturation coefficient determined repeatedly on the same specimen. The saturation coefficients of samples *a* and *b* of brick E appear subject to a systematic difference since the average for sample *b* exceeds *a* by 0.023 and without exception all specimens of the *b* halves had higher saturation coefficients than the corresponding *a* halves.

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[7] W. N. Thomas, "Experiments on the Freezing of Certain Building Materials," *Technical Paper No. 17*, Building Research Board, Department of Scientific and Industrial Research (1938).

Stull and Johnson[5] found that the porosity of bricks A, B, C, and D determined by the gas-expansion method was approximately the same as that calculated from the absorption by 5-hr. boiling, but for brick E, the porosity by the gas-expansion method was appreciably higher than that calculated from the water absorption. This means that not all of the pores were filled with water even after 5 hr. of boiling.

Bricks which, like E, show a significant difference between the porosity by the gas-expansion method and by the boiling method usually give variable results in repeated absorption tests, being sensitive to small differences in time and temperature of cooling. Since the determinations on the two halves of brick E were made at different times, it is believed that the difference in saturation coefficients for the two halves represent differences in method rather than in specimens.

Discussion of Methods of Freezing and Thawing.—The suppression of all controllable variables except the temperature of freezing and the rate of thawing was attempted in the conduct of these tests. Freezing of both samples of the same brick took place at the same time and thawings were made simultaneously in the same tank. The two curves in Fig. 1 could be essentially duplicated at a single temperature of freezing (−20 F.) if one of the samples had been suitably insulated. The preliminary tests on 2-in. brick cubes demonstrated that, under the conditions of exposure then used, 5 hr. were required to completely freeze the specimens at −20 F. Data published by Thomas[7] showed that after freezing has been accomplished, a further drop in temperature had little destructive effect, so it appears that the rate of freezing is more important than the final temperature in its effect on disintegration.

Aside from temperature control, the method of freezing and thawing here used may be considered as intermediate between methods 1 and 3 described in the 1938 paper.[2] The time rate of freezing in the chamber for an air temperature of 18 F. is also given in Fig. 1. In method 1 the saturation increase was checked after every third cycle while method 3 permitted saturation to increase without being checked.

Effect of Freezing on Brick Samples.—The first and only failure by method *a* (20 F.) in the sample of dry-pressed clay brick, (brick A, Table I), was brick 12A, which failed at 45 cycles. There were 17 failures by method *b*, the first of which was brick 4A, failing at 21 cycles. In the case of the bricks which did not fail, little choice is evident between the severity of the two methods in causing loss in weight. Method *a* caused a greater loss in weight than did method *b* on the three specimens that survived 50 cycles by both methods. Of the 17 specimens that survived 25 cycles by both methods, eight gave greater losses by method *a*, eight by method *b*, and one gave the same loss for both methods. From this it is concluded that freezing at −20 F. is more severe in disrupting or breaking this type of brick, but there is little difference between the two freezing temperatures in causing surface damage.

The "unmarketable salmon" side-cut shale brick (brick B, Table II) is so lacking in resistance that differences in severity of freezing-and-thawing methods tend to become obscured if they are based solely on the number of specimens surviving a given number of cycles. There were 15 failures by method *a* and 16 failures by method *b* in the 50 cycles. However, method *b* (−20 F.) caused a greater loss in weight at the same number of cycles or failure in fewer cycles than method *a* (20 F.), in 18 of the 20 samples, the two exceptions being the pairs 4B and 18B.

The soft-mud clay brick (brick C, Table III) showed no significant difference between the two methods. As stated in the previous paper, "the C samples are characterized by being very nonresistant at one end of this distribution (of saturation coefficients) and highly resistant at the other." The C specimens are soft mud bricks of a type which loses weight by surface dusting. There were seven failures by each method at 50 cycles, and, as in the case of brick A, there was no differentiation between methods *a* and *b* in causing weight loss. Compressive strengths were determined on specimens 1C to 10C for both samples *a* and *b* after completion of 50 cycles of freezing and thawing. The *a* samples averaged 2025 and the *b* samples 1760 psi. This, however, is of doubtful significance.

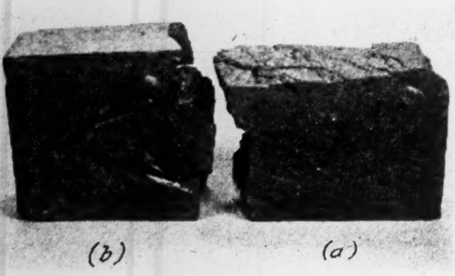

FIG. 2.—The Two Halves of Specimen 18D, Showing the Two Types of Failure Observed; (*a*), frozen at 20 F. and (*b*) at −20 F.

Brick D (Table IV) of side-cut shale represents a rather laminated side-cut brick of high strength in comparison with the types hitherto considered. Such bricks tend to fail by disruption and breakage. The relative effectiveness of low-temperature freezing in producing breakage is strikingly illustrated by this sample. In 19 cycles all 20 specimens of brick D were destroyed when frozen at −20 F., while but two specimens out of 20 were destroyed by the same number of cycles at 20 F. Figure 2 illustrates pair 18D. Specimen *a* of this pair had undergone 17 cycles at 20 F. and had progressively spalled beginning at the eighth cycle. No single spall was thicker than ⅛ in. Sample *b* broke suddenly on the seventh cycle. These two types of failure are characteristic of brick D. At the higher temperature progressive spalling occurred on one or both faces and at the lower temperature shattering and

TABLE V.—COMPARISON OF EFFECT OF FREEZING AT 20 F. AND AT −20 F. ON A SAMPLE OF AN END-CUT CLAY BRICK (BRICK E).

Specimen Number of Whole Brick	Saturation Coefficient of Half Bricks		Loss in Weight as Percentage of Original Dry Weight, or Number of Cycles to Failure (F−c) or Breakage (B−c)					
			25 cycles		34 cycles		50 cycles	
	Sample *a*	Sample *b*.	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.	Sample *a*, 20 F.	Sample *b*, −20 F.
1E	0.803	0.824	0.42	0.33	0.60	F27c	1.36	....
2E	0.817	0.847	F24c	0.95	....	1.06	...	2.43
3E	0.818	0.837	0.31	0.47	0.39	0.47	0.55	B45c
4E	0.810	0.845	0.42	0.21	0.74	0.21	1.05	0.48
5E	0.820	0.836	0.24	F17c	0.31	....	0.55	....
6E	0.830	0.855	2.74	0.62	2.99	0.71	4.48	B41c
7E	0.826	0.844	0.40	B19c	0.49	....	0.79	....
8E	0.827	0.840	0.24	F14c	0.41	....	0.74	....
9E	0.823	0.846	0.48	B23c	0.95	....	1.34	....
10E	0.822	0.855	F13c	1.06	....	1.74	...	F50c
11E	0.831	0.853	0.47	0.44	0.47	0.66	0.74	B39c
12E	0.825	0.861	F7c	F13c	....	....	....	....
13E	0.838	0.853	1.97	B21c	2.52	....	F46c	....
14E	0.838	0.856	0.25	0.50	0.58	0.57	0.92	B49c
15E	0.851	0.879	F24c	B25c	....	....	....	....
16E	0.860	0.876	F12c	6.30	....	F27c	....	....
17E	0.849	0.860	F9c	0.29	....	0.48	....	B43c
18E	0.847	0.885	F25c	3.21	....	F29c	....	....
19E	0.865	0.892	F24c	B24c	....	....	....	....
20E	0.865	0.880	F2c	F5c	....	....	....	....
Avg.	0.833	0.856						

breakage took place without preliminary surface spalling.

Brick E (Table V), an end-cut clay brick, resembles brick D in its tendency to exhibit two types of failure (progressive spalling at 20 F. and shattering at −20 F.). For the first 25 cycles the same number of failures occurred for both temperatures. Freezing at −20 F., however, was more severe than that at 20 F. during the last 16 cycles.

TABLE VI.—NUMBER OF SPECIMENS IN EACH SAMPLE PASSING 25, 34 AND 50 CYCLES OF FREEZING AND THAWING USING 10, 3 AND 1 PER CENT LOSS IN DRY WEIGHT AS THE CRITERION OF FAILURE FOR FREEZING AT 20 F. AND AT −20 F.

Brick	10 per cent Loss in Weight		3 per cent Loss in Weight		1 per cent Loss in Weight	
	20 F.	−20 F.	20 F.	−20 F.	20 F.	−20 F.
25 CYCLES						
A	20	17	20	17	17	12
B	9	7	6	6	0	1
C	13	14	12	12	11	11
D	18	0	16	0	14	0
E	11	11	11	9	9	8
34 CYCLES						
A	20	10	19	10	12	6
B	7	7	5	4	0	0
C	13	13	11	11	9	11
D	17	0	16	0	13	0
E	11	8	11	8	9	6
50 CYCLES						
A	19	3	18	3	6	1
B	5	4	3	2	0	0
C	13	13	11	11	5	6
D	11	0	8	0	8	0
E	10	2	9	2	6	1

The results in Tables I to V are summarized in Table VI, which shows the number of brick surviving 25, 34, and 50 cycles of freezing and thawing at 20 F. (method *a*) and −20 F. (method *b*). This table also shows the number of bricks from each sample surviving at 25, 34, and 50 cycles when the criterion of failure is changed to 3 per cent and 1 per cent loss of the original dry weight, respectively. These are the criteria of failure used in the Society's Tentative Method of Freezing-and-Thawing Test of Brick (C 67 – 39 T)[8] for grades MW and SW, respectively.

Comparison of the data reported in Tables I to V with the data reported in Table VI of the 1938 report, a summary of which appears in Table VII of this report, leads to the following conclusions:

1. Freezing and thawing at 20 F. under the conditions stated is not as severe as any of the freezing methods using a cold room of the type and temperature described in 1938. The least severe of the 1938 methods (method 4) passed method *a* at 12 cycles and at 50 cycles had caused 56 failures out of a possible 100 failures compared with 42 failures for method *a*.

TABLE VII.—NUMBER OF SPECIMENS IN EACH SAMPLE OF BRICKS A, B, C, D AND E PASSING 50 CYCLES OF FREEZING AND THAWING WHEN TESTED BY METHODS 1 TO 7, METHOD *a* (20 F.) AND METHOD *b* (−20 F).

The criterion of failure is breakage or 10 per cent loss. Twenty specimens constitute a sample.

Brick	Method 1	Method 2	Method 3	Method 4	Method 5	Method 6	Method 7	Method *a*	Method *b*
A	20	20	8	1	0	11	0	19	3
B	1	3	0	7	0	0	2	5	4
C	10	9	5	9	10	10	12	13	13
D	3	1	0	14	0	0	9	11	0
E	2	4	0	13	2	3	8	10	2
Totals	36	37	13	44	12	24	43	58	22

2. The results of freezing and thawing at −20 F. under the conditions stated were exceeded in severity at all cycles only by method 3* of the 1938 series.[2] Method 5 was less effective up to the thirty-fourth cycle and thereafter exceeded the low-temperature method. Method 6 was more effective than the −20 F. method up to the thirty-fourth cycle and thereafter matched it. Method 6 is "method B" of the Society's tentative method C 67 – 39 T.[8]

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[8] 1939 Book of A.S.T.M. Standards, Part II, p. 930.

* The specimens tested by method 3 were continuously wet and thus an unchecked increase in degree of saturation was permitted.

The two preceding conclusions were based on totals for all five types of brick. Table VII presents a comparison of the effectiveness of methods 1 to 7 of the 1938 report[2] with the two methods using controlled temperature. It will be noted that method *b* is less severe than method 3 except on brick A (brick D showed no difference). It may be concluded that an increase in degree of saturation build-up is more of a factor than lowering the freezing temperature.

Conclusion 1 in the 1938 paper was, "The nature of the brick influences the relative effect of differences in the methods of test." This statement was based on the effect of variations in test methods which influenced the relative degree of water saturation of the specimens while being frozen. The same statement applies to the effect of temperature according to these data.

SUMMARY AND CONCLUSIONS

This paper reports the effect on the resulting disintegration of two comparable samples of half bricks, of freezing and thawing for 50 cycles using freezing temperatures of 20 F. and −20 F., respectively. These two comparable samples were formed by breaking a single sample of whole bricks into half bricks. The sample included bricks formed by the dry-press, soft-mud, and stiff-mud processes and was comparable with previous samples tested in 1938.

From the data herein reported the following conclusions appear to be justified:

1. Freezing at −20 F., as compared to 20 F., greatly increased the amount of disintegration and breakage on three of the five types of bricks tested. One additional type of brick was so lacking in resistance that there was little difference in the number of specimens surviving 50 cycles by each method, but in 18 of the 20 cases freezing at −20 F. produced failure at fewer cycles or a greater loss in weight at the same number of cycles than did freezing at 20 F. The fifth type of brick showed little difference between the two methods. The nature of the brick influenced the degree of disintegration resulting from freezing at −20 F. and 20 F.

2. Freezing at −20 F. tended to produce failure by breaking, while progressive loss from the surfaces of the bricks was the usual type of disintegration resulting from freezing at 20 F. Where failure did not occur by breaking or disruption, no consistent difference in the severity of the two temperatures was observed as judged by rate of loss in weight.

3. The rate of freezing was much affected by the temperature to which the specimens were subjected under the conditions of test. At 20 F. the center of a half brick remained at 32 F. for 3 hr. while at −20 F. this time was reduced to 15 min. It is considered that rate of freezing rather than temperature is the effective factor in influencing rate and amount of disintegration. The effect of difference in rate of thawing entered into these tests but was not evaluated.

4. Freezing in an open chamber with an air temperature varying from 20 F. to 12 F. exceeded in severity the effect of freezing in rubber bags at −20 F. when the open chamber freezing permitted unchecked increase in saturation. With an air temperature of 18 F., the bricks required approximately 40 min. to complete freezing. Freezing at 20 F. in rubber bags was less effective than any of the methods using an open-air freezing chamber.

PROPERTIES OF LIMES AND LIME MORTARS

By Howard R. Staley[1]

Synopsis

This paper presents the results of observations made upon hydrated-lime pastes and hydrated lime - sand mortars. Tests on the pastes include plasticity and sedimentation or bleeding measurements. Investigation of the characteristics of the hydrated lime - sand mortars include, mixing-water requirements, density of the freshly mixed mortars, flow after suction, and sand-carrying capacity. Three methods are used for determinine sand-carrying capacity of which the extrusion machine appears to be most consistent.

Derived data include the immobile water in the pastes, apparent mean particle size and apparent surface area of the limes.

Purpose:

The purpose of this investigation was to study the physical characteristics of the hydrated limes used and the effect of these physical characteristics upon mortars made with them.

Materials:

Seven lime hydrates were used, three being high-calcium and four dolomitic. For purposes of calculation of volumetric proportions, the weight of hydrated lime was taken as 45 lb. per cu. ft.

Lime Hydrate	Type	Apparent Specific Gravity in Water
No. 1	High-calcium	2.197
No. 2	High-calcium	2.087
No. 3	High-calcium	2.148
No. 4	Dolomitic	2.439
No. 5	Dolomitic	2.435
No. 6	Dolomitic	2.137
No. 7	Dolomitic	2.144

The sand used weighed 95 lb. per cu. ft. dry and loose, with an apparent specific gravity of 2.65 and had the following sieve analysis:

Sieve	Retained on Sieve, per cent
No. 100	90.50
No. 48	69.83
No. 28	32.32
No. 14	11.65
No. 8	5.95
No. 4	5.00
Fineness modulus	2.15

Standard Ottawa sand was used for the water-retention test.

Cambridge tap water was used for all mixing.

Proportioning:

All proportioning was done on a weight basis. Where translations to volume were made, they were made using the weights given above.

Mixing:

Mixing for the water-retention test was performed according to A.S.T.M.

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Standard Specifications for Masonry Cement (C 91 – 40).[2]

In mixing mortars for the density measurements the procedure given below was followed. The hands were covered with rubber gloves during mixing.

1. Add water to nonabsorbent bowl.
2. Add dry lime to water and mix until wetted.
3. Add 1500 g. of sand, then mix for 3 min.
4. Allow to rest 1 min.
5. Mix 1 min.
6. Take flow.
7. Return material used in flow test to bowl and remix for 30 sec.
8. Measure density.

The mixes for sand-carrying capacity were made in a flat mixing pan using a trowel for mixing. The dry materials were thoroughly mixed together until of uniform color after which water was added to yield the proper consistency. Flow was then taken at this time. Fifteen minutes after the first addition of water, the material was placed in the cylinder for extrusion.

The pastes for sedimentation tests were mixed with a mechanical mixer. Time was counted from the completion of the addition of the dry lime hydrate to the water in the mixing bowl. Mixing was carried out at a relatively slow speed. The slurry was mixed 2 min., allowed to rest 3 min., then remixed for 2 min. after which it was immediately transferred to a beaker for sedimentation measurement.

TEST METHODS

A standard 10-in. flow table mounted on a concrete base was used for all flow measurements. Water-retention tests were made using the Rogers apparatus following A.S.T.M. procedure C 91 – 40.

Sand-carrying capacity measurements were made using the extrusion device.[3] The proportion of sand to lime was varied by $\frac{1}{4}$ part by volume until a proportion was obtained which could not be extruded without excessive pressure. The proportion $\frac{1}{4}$ part lower than this was termed the sand-carrying capacity.

A test for sand-carrying capacity developed by J. H. Pettit of Philadelphia, Pa., was also used. This test consists of placing 40 ml. of water and 50 g. of sand in a 100-ml. graduate, then adding lime hydrate in small increments until cessation of flow after vigorous shaking. The sand-carrying capacity is given by the relation

$$S = \frac{K}{W}$$

where

$S = \frac{\text{volume sand}}{\text{volume lime}}$,

W = weight of lime added in grams, and

K = constant = 103.

Density of the freshly-mixed mortars was obtained by weighing portions of the mortar in a rigid brass container 3 in. in diameter and 6 in. high.

The Emley plasticimeter was used to determine the plasticity of the limes after soaking for 1 and 24 hr.

Apparent specific gravities of the limes were determined with a pycnometer bottle. Water was used as the liquid for the determination.

Sedimentation measurements were made by observing the subsidence of the paste surface with a microscope using the micrometer screw to measure the amount of drop. The surface of the paste was covered with a film of water and then a film of kerosine to prevent

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[2] 1940 Supplement to Book of A.S.T.M. Standards, Part II, p. 1.

[3] Walter C. Voss, "Lime Characteristics and Their Effect on Construction," Symposium on Lime, p. 103, Columbus Regional Meeting, Am. Soc. Testing Mats. (1939). (Symposium issued as separate publication.)

TABLE I.—DATA ON MORTARS.

Sand, per cent	Water, per cent[a]	Flow, per cent	Density, g. per cu. cm.	Voids Filled, per cent[b]
Lime No. 1—High Calcium				
70.0	25.50	105	1.890	200.0
80.0	21.50	125	1.980	136.8
82.5	19.50	109	1.988	119.3
85.0	18.50	110	1.989	106.6
87.5	18.00	110	2.000	99.0
90.0	17.90	112	2.000	88.0
92.5	18.25	106	1.988	83.7
95.0	19.23	122	1.978	81.1
97.5	19.50	101	1.943	75.6
Lime No. 2—High Calcium				
70.0	31.00	105	1.840	224.0
80.0	23.00	105	1.958	145.5
82.5	22.00	109	1.982	132.0
85.0	21.25	114	2.004	119.5
87.5	19.50	107	2.041	104.2
90.0	19.00	107	2.060	94.5
92.5	18.50	116	2.078	85.4
95.0	19.80	120	2.062	83.7
97.5	20.50	119	2.058	79.8
Lime No. 3—High Calcium				
70.0	30.40	115	1.870	216.0
80.0	22.50	111	1.960	142.0
82.5	22.00	104	1.962	131.0
85.0	21.50	104	1.970	119.8
87.5	20.50	115	1.992	107.8
90.0	19.60	111	1.999	96.0
92.5	19.00	111	2.003	87.1
95.0	20.00	121	1.979	84.1
97.5	21.50	117	1.962	83.2
Lime No. 4—Dolomitic				
70.0	26.00	109	1.958	195.0
80.0	21.50	115	2.010	132.8
82.5	20.50	112	2.018	120.0
85.0	18.10	107	2.025	102.0
87.5	18.30	105	2.035	95.6
90.0	18.38	115	2.030	88.8
92.5	18.50	107	2.028	84.4
95.0	19.20	115	1.987	80.2
97.5	20.50	112	1.961	79.0
Lime No. 5—Dolomitic				
70.0	26.65	115	1.927	198.8
80.0	22.00	119	2.015	135.0
82.5	20.30	114	2.030	119.5
85.0	18.75	110	2.035	104.8
87.5	18.40	108	2.030	96.3
90.0	18.50	112	2.028	89.5
92.5	18.75	113	2.015	84.5
95.0	19.25	110	1.982	80.2
97.5	20.50	112	1.958	79.2
Lime No. 6—Dolomitic				
70.0	31.50	108	1.820	232.0
80.0	26.50	112	1.905	160.1
82.5	25.00	112	1.937	144.2
85.0	23.60	110	1.955	129.0
87.5	22.50	114	1.971	116.0
90.0	21.50	113	1.980	103.7
92.5	20.25	113	1.990	93.0
95.0	20.0	117	1.980	84.1
97.5	20.0	110	1.952	77.7
Lime No. 7—Dolomitic				
70.0	27.50	112	1.890	211.0
80.0	23.00	110	1.969	144.2
82.5	22.00	109	1.979	131.0
85.0	21.50	110	1.990	120.0
87.5	21.00	115	2.000	109.7
90.0	20.50	112	2.002	100.0
92.5	20.15	113	2.005	91.4
95.0	20.00	118	2.003	84.1
97.5	20.50	114	1.979	79.6

[a] Percentage of dry materials in batch.

[b] Percentage of voids filled in the dry-and-loose sand in the batch.

evaporation and carbonation. Sedimentation of the surface of the paste was noted by focusing on a particle then refocusing on the same particle after sedimentation. Several measurements were taken over a period of 30 min., and the rate of settling was averaged over this period. All measurements of sedimentation were made in a constant-temperature room which was maintained at 70 F.

TEST RESULTS

Data on the various mixes of mortars made are given in Table I. Nine mixes were made for each lime, varying in sand from 70 to 97.5 per cent by weight of the dry materials. The percentage of water required to produce a definite consistency (per cent by weight of the dry materials), the percentage flow, and the density of the freshly-mixed mortars are given for each of the mixes. The percentage of voids in the dry-and-loose sand filled by the paste volume was calculated using the apparent specific gravities and unit weights given previously. These mixes were made to obtain the variation in water requirements for the various proportions and to examine the manner in which the density of the freshly-mixed mortars varied.

Water Requirements for Mortars:

Referring to Fig. 1, it is seen that the percentage of mixing water decreased to a minimum with increasing sand content and increased again beyond the minimum, with further increases in sand content. For convenience, only the values above 80 per cent of sand are plotted. The end-point for 100 per cent sand was determined by making a flow test of sand and water, and while this is difficult to do, the value given is as near correct as is possible to determine by this method. The voids in this quantity of dry-and-loose sand were approximately 82.0 per cent filled with water at this point. At the commonly used volumetric proportion of 2.5 parts of sand (84.0 per cent by weight), the variation in the amount of mixing water required for the seven limes is 5.4 per cent; at 3.0 parts sand (86.4 per cent by weight), the variation in water requirements is 4.8 per cent. Beyond 89.5 per cent sand (or 4.0 parts sand by volume) the variation in mixing-water requirements decreases quite rapidly. Except for the very lean mixes, the mortars made with lime No. 6 have the highest water requirements and those with lime No. 1 the lowest. The rate of decrease in mixing water with increasing sand content is approximately the same between 80 and 90 per cent sand, except for the mortars made with limes No. 3 and No. 7. The mortars made with lime No. 3 do not follow the same tendencies shown for the others in many respects and those made with lime No. 7 are at variance with the others only in respect to change in mixing-water requirements. Except for lime No. 3, one could qualitatively classify these limes for sand-carrying capacity by merely a consideration of the placement of the curves at say 85 to 90 per cent sand, that is, the higher the water requirement for mixing water, the higher the sand-carrying capacity. This order might be somewhat changed if a different sand were used, but it is believed that it would be approximately the same for any graded sand. The mortars made with lime No. 7 show the least change in water requirements of all the limes considered. The differences in water requirements for the limes is indicative of different structures in the pastes themselves.

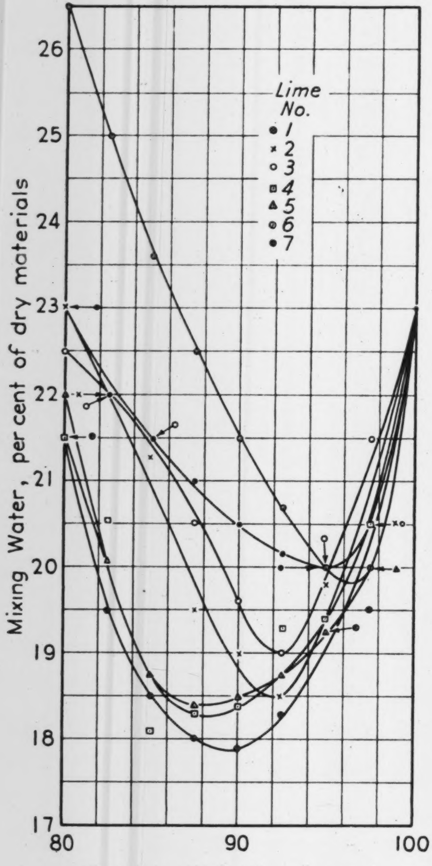
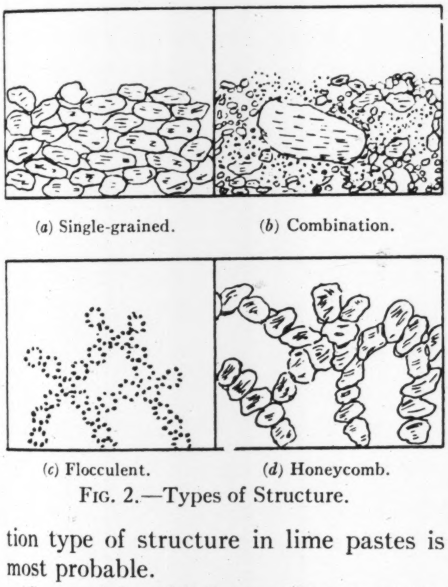


FIG. 1.—Variation in Mixing Water for Mortars.

An indirect measure of the degree of dispersion or the development of a flocculent, honeycomb, or single-grained structure in a lime paste, is the amount of water required to produce a certain consistency. The development of a type of structure in the lime paste depends largely upon the physical characteristics of the particles. These physical characteristics may be due to size and shape of particle, surface activity or adsorption, the porosity of the lattice structure of the compound (similar to bentonite), or to all of these combined. The types of structure indicated are illustrated in Fig. 2. Loose sand is a typical example of single-grained structure while many natural clays are examples of the other types and for the combination type shown. The existence of the combination type of structure in lime pastes is most probable.


(a) Single-grained. (b) Combination.

(c) Flocculent. (d) Honeycomb.

FIG. 2.—Types of Structure.

As sand is added to a lime paste in increasing amounts the presence of the granular particles of the sand tends to produce a single-grained structure, thus reducing the water requirements to make a mortar of a certain consistency. Beyond the point of minimum water requirement the dispersion of the lime particles becomes so great that increased water is necessary to create a large enough volume of paste to lubricate the mass properly to the desired consistency.

The type of structure in the lime paste will affect the workability of the mortar to a large extent. For example, limes No. 6 and No. 7 have approximately the same apparent specific gravity in water. The ability of a paste made with lime No. 6 to hold the same amount of sand in suspension and still be cohesive enough to have the same flow with a greater water content than lime No. 7, must be due to forces acting between the lime particles in the paste. In other words, there is more coherence and effective arch action of the lime particles present in the paste with lime No. 6 than with lime No. 7. The difference in the slope of the two curves, shown in Fig. 1, representing change in water requirements for limes No. 6 and No. 7, would indicate that the structure in the paste of lime No. 7 was more of the honeycomb type, while that in lime No. 6 was more of the flocculent type.

Density of Mortars:

Examination of the data given in Table I shows that the density of the mortars increased to maximum at or near the point of minimum water requirement as would be expected. For limes Nos. 1, 4, and 5, maximum density of the mortars occurred when the voids in the quantity of dry-and-loose sand used were approximately 100 per cent filled. For the other limes, maximum density occurs with the calculated paste volume filling the voids in the sand 85.3 to 93 per cent. If one calculates the density of the pastes it is almost always found that the calculated value for density will be higher than that obtained by actual measurement due to the fact that air is always occluded in the mortar.

Sand-Carrying Capacity:

Sand-carrying capacity is a function of the cohesiveness and the plasticizing ability of the paste, when mixed with sand. It depends directly upon the ability of the paste to lubricate the mass and to cohere and hold the sand in suspension while the mortar is at rest, or during transportation or deformation. Harsh-working mortars are the result of using a matrix that does not possess the characteristics given above, or the "suspending capacity" of the paste is exceeded by using an excessive amount of sand.

The results of the sand-carrying capacity tests are given in Table II.

Referring to the values given in Table II, there is good agreement in the extrusion test and the Pettit test except for limes Nos. 3 and 7. The values for the extrusion machine test and the void method agree closely except for lime No. 3. The disagreement for lime No. 3 in the three tests cannot be adequately explained. An explanation of the disagreement for lime No. 7 with the Pettit test is that the structure built up by the lime particles in the richer paste in the Pettit test is different than for the other limes. This is suggested by the slope of the curve in Fig. 1 showing a decreased rate of change in water requirement for the richer mortars.

TABLE II.—SAND-CARRYING CAPACITY.[a]

Lime	Extrusion Machine		Pettit Test, parts by volume	Void Method	
	Parts by Volume[b]	Sand, per cent		Parts by Volume	Sand, per cent
No. 1........	2.75	85.3	2.75	2.88	85.8
No. 2........	3.50	88.0	3.79	3.30	87.4
No. 3........	2.50	84.0	3.33	3.42	87.8
No. 4........	2.75	85.2	2.78	2.78	85.4
No. 5........	2.75	85.2	2.78	2.71	85.1
No. 6........	4.00	89.5	3.96	3.96	89.5
No. 7........	3.75	88.7	3.43	3.67	88.6

[a] Sand-carrying capacity $= \frac{\text{Volume sand}}{\text{Volume lime}}$.

[b] For extrusion tests nearest quarter part taken.

The results obtained with the Pettit test can be explained on the following basis: In making the test, the force causing deformation or flow, when the graduate containing the sand-lime-water mix is turned up, is that of gravity. At cessation of flow, the structure of the mass is strong enough to resist the force of gravity tending to deform it. In other words, the yield point of the mortar is large enough to resist the gravitational force and in effect this test gives roughly the point of zero deformation under this force. It is known that all pastes having the same fluid content do not have the same yield point or the same resistance to deformation. In fact, to obtain exactly zero deformation or any constant amount of deformation for various pastes under a definite load or force, the pastes might have widely varying fluid content. This is shown by the results of the Pettit test. This is also corroborated by the consistency test for the Emley plasticimeter as all limes do not require the same amount of water to make a paste of standard consistency. The sand-carrying capacity of a paste is measured in the extrusion device by its resistance to plastic flow. The equation of plastic flow can be applied to this as well as any process of extrusion.

With the extrusion device, the sand-carrying capacity is arbitrarily defined as that sand-paste mixture, of a definite consistency, which shows little or no resistance to extrusion. At the proportion of sand-carrying capacity, the paste proportion of the mix may be of widely varying water contents, with different materials, to produce a definite consistency in the mortar. In the Pettit test the same principles are involved but the approach is from a slightly different angle as the force causing deformation is much less.

Roller[4] has shown that the yield value of pastes of any one material decreases with increasing water contents and that for cement mortar the yield value decreases with increasing sand content with a corresponding decrease in water content. It might be that the coefficient of renitence K, as determined by Roller, is a measure of the effective plasticizing ability of pastes in mortars, as the values of K obtained by him for a 1:2 and 1:2.6 cement-sand mortar show a sharp increase from the 1:2 mortar to the 1:2.6 mortar, indicating the possibility of a minimum value in this range of proportions. The sand-carrying capacity of portland cements, as measured by the extrusion device, is commonly around 1:2 to 1:2¼, cement and sand by volume, which is in the range of the values used in Rollers' investigation.

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[4] Paul S. Roller, "Plastic Flow of Dispersions and a New Approach to the Study of Plasticity," *Journal of Physical Chemistry*, Vol. 43, No. 4, April, 1939, p. 457.

The percentage of sand given under void method is the percentage of sand, by weight of the dry materials, obtained when the voids in the dry-and-loose sand in the mortar are 105 per cent filled with paste. Figure 3 shows the variation of percentage fullness of voids in the sand, plotted against percentage of sand by weight of the dry materials in the batch. The intersection of the curves and the horizontal line at 105 per cent gives the sand percentage at sand-carrying capacity. The sand-carrying capacity of a lime may be found by the void method as follows:

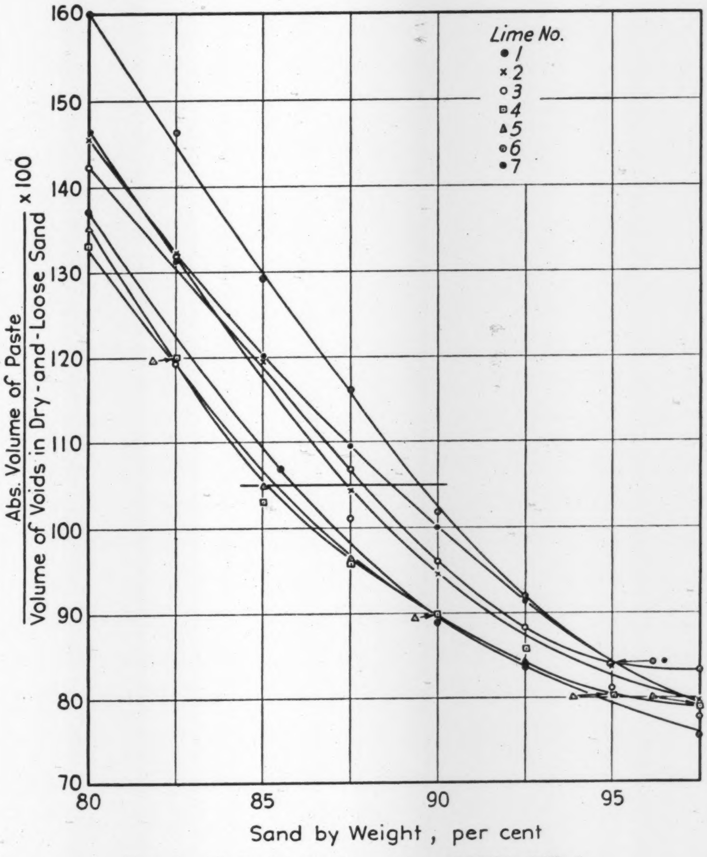

FIG. 3.—Variation in Percentage of Voids Filled.

Take a proportion of sand and lime 85:15 by weight and determine the water necessary to give a flow of 110 to 115 per cent. Make the same determination for a mix of 90:10 sand and lime, using the mixing procedure previously given for both mixes. Determine the percentage of voids filled with paste in the dry-and-loose sand in both batches, then plot these values against sand percentage, assuming a straight-line variation. Since the sand-carrying capacity occurs when the sand voids are filled 105 per cent, the value for this percentage of sand can readily be determined from the plot. The approximate mixing water for the point can also be found by plotting the percentage of mixing water at the end points, then taking the percentage water at the value for the sand percentage found.

TABLE III.

Lime	1 Hour	Plasticity Figure, 24 hr. soaking	Flow After Suction, per cent
No. 1	117	140	89.0
No. 2	130	139	94.0
No. 3.	129	226	88.0
No. 4	127	134	91.0
No. 5	126	140	89.0
No. 6	300	386	95.0
No. 7	237	280	95.0

There appears to be no exact relationship between maximum density or minimum water requirement and sand-carrying capacity. This sand percentage at sand-carrying capacity is always lower than that at the point of maximum density or minimum water requirement for the lime-sand mortars.

Plasticity and Water-Retention:

Values for the plasticity figures and flow after suction are given in Table III.

Plotting the values given in Table III shows no evident relation between plasticity and flow after suction. The values given for 24 hr. plasticity and flow after suction are plotted against sand-carrying capacity, as measured by the extrusion machine, in Figs. 4 and 5. Figure 4 shows that no exact relation exists between plasticity and sand-carrying capacity. It is indicated here that the mere fact that a lime has a plasticity of over 200 does not signify that it is a better lime for use in mortar than one which does not have a figure that high. The two limes with the highest plasticity figures do show the highest sand-carrying capacity, but of the other five, the only one which has a plasticity of over 200, has the lowest sand-carrying capacity.

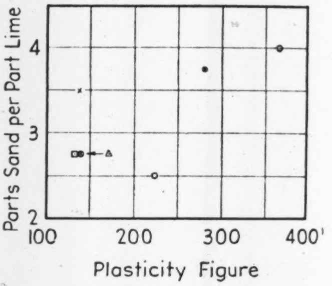

FIG. 4.—Relation of Sand-Carrying Capacity and Plasticity Figure.

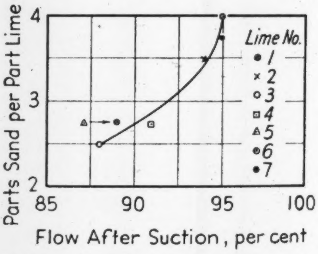

FIG. 5.—Relation of Sand-Carrying Capacity and Flow After Suction.

A much better relation exists between flow after suction and sand-carrying capacity as shown in Fig. 5. It is not known whether this relation would exist for other than lime mortars, but it is intended to enlarge the scope of this work to include other mortars at a later date. However, for lime mortars, the flow-after-suction test is evidently a better indicator of the value of a lime for use in mortar than is the plasticity test.

Sedimentation Tests:

Some sedimentation, water-gain, or bleeding occurs with all mortar and concrete mixes because a certain amount of excess water is always present. The amount of bleeding depends upon the ability of the cementitious material to hold the water in approximately the same relative position in the mixture at all times. A certain amount of bleeding is beneficial in that the density of the mass is increased. On the other hand, a cementitious material with a high bleeding rate is apt to produce harsh-working mixes because of the segregation of the water. The chief function of the water is to lubricate the mass of concrete or mortar and unless this water is held in proper position long enough to perform this function, a large amount of bleeding occurs with a resultant loss in workability in the mix. In the case of concrete, the force causing sedimentation of the solids or bleeding is the force of gravity. In the case of masonry mortar the gravitational force is probably negligible due to the fact that the suction of the masonry unit is usually far greater and the thickness of the mortar layer is small. However, it is possible to make a comparison of quantitative measurements on pastes as the fundamental characteristics of the paste governs the relative amount of bleeding in the paste or the mortar.

In order not to make this paper too lengthy, data for the sedimentation tests are not presented. It is probably sufficient to say that for the limes having the lower immobile-water factors, the water in the pastes for sedimentation tests varied from 50 to 70 per cent of the weight of the paste. For those limes with the higher immobile-water factors, the water in the pastes varied from 60 to 80 per cent of the weight of the paste. No attempt has been made to include a discussion of bleeding, but for a complete discussion the reader is referred to a paper by T. C. Powers.[5] The formula used for calculations was derived by Powers. It was developed from Poiseuille's law governing hydraulic flow through small capillaries which is identical in form with D'Arcy's equation governing the flow of water through granular beds:

$$Q = \frac{g \cdot (w - w_i)^3 \cdot (d_c - d_f)}{K_o \cdot n \cdot c \cdot \sigma^2}$$

where

Q = the rate of bleeding per unit of gross cross-sectional area of the sample, milliliters per square centimeter per second, identical with the rate of subsidence of the surface of the paste, in centimeters per second,

g = 980 centimeters per second per second,

w = the absolute volume of water per unit volume of the paste,

w_i = the immobile-water factor or the volume of immobile water per unit volume of mix,

d_c = the density of the solid in grams per cubic centimeter,

d_f = the density of the fluid in grams per cubic centimeter,

n = the viscosity of the fluid in grams per centimeter-second,

c = the absolute volume of solid per unit volume of paste,

σ = the specific surface of the solids in square centimeters per cubic centimeter, and

k_o = a constant, identical with the constant in Kozeny's equation for general law of streamline motion through granular beds. The value of k_o = 5.0 was used for these tests.

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[5] T. C. Powers, "The Bleeding of Portland Cement Paste," published by the Portland Cement Assn. (1939).

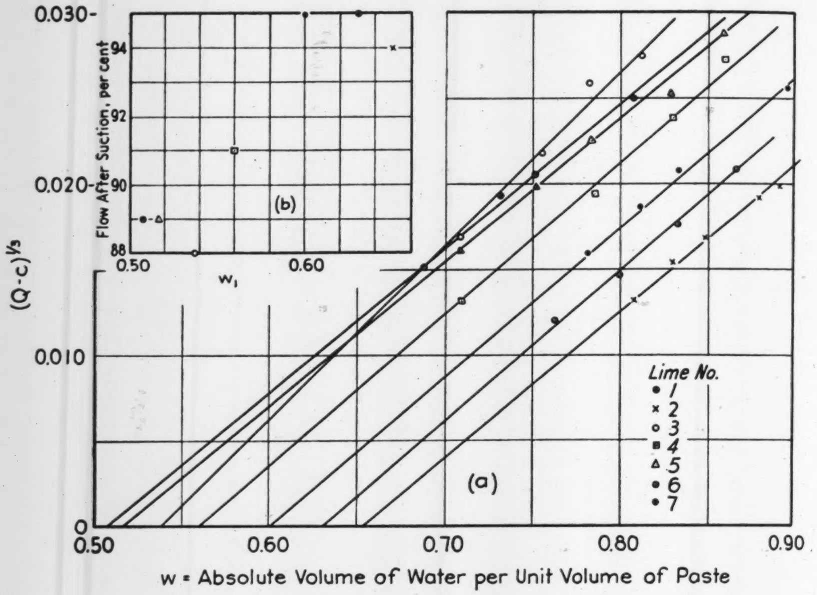

FIG. 6.—Comparison of Sedimentation Tests.

The above equation can be written,

$$(Q.c)^{1/3} = K^{1/3}\cdot(w - w_i)$$

where $K = g\cdot(d_c - d_f)/k_o\cdot n\cdot\sigma^2$

If a graph is made plotting $(Q\cdot c)^{1/3}$ against w, a straight line results which crosses the w_i axis at a point equal to w_i and having a slope equal to $K^{1/3}$

The immobile-water factor w_i was introduced by Powers to account "for the liquid held stationary with respect to the solid surfaces by minute imperfections in the surfaces or by variations in the channels which might give rise to a condition analogous to backwater or eddies.... With finer materials ... liquids might be held in appreciable quantities by chemical and physical forces which may vary through a wide range depending on the natures of the solid and liquid brought together.... there is reason to believe that differences in the plasticity of pastes made with mineral powders and water, differences not accounted for by the differences in fineness, might be related to w_i. It seems likely that with other factors equal, a material with a high w_i value should show more 'fatness' than one with a low w_i value."

Figure 6 (*a*) shows the variation of $(Q\cdot c)^{1/3}$ with w. The values for w_i recorded in Table IV were obtained from this plot. The highest values for w_i are shown for limes Nos. 2, 6, 7, and 4; No.

TABLE IV.

Lime	w_i, absolute volume of water in unit volume of paste	Apparent Surface, sq. cm. per cu. cm.[a]	Apparent Particle Diameter, μ	Thickness of Film of Immobilized Water, μ	Excess Water[b]	
					Milliliters per gram of Lime	Total Water, per cent
No. 1	0.508	6460	9.3	0.78	0.77	62.1
No. 2	0.652	6270	9.6	1.04	0.54	34.2
No. 3	0.538	4775	12.5	1.13	0.88	62.0
No. 4	0.560	4895	14.6	1.14	0.73	58.4
No. 5	0.517	4425	13.5	1.15	0.79	64.0
No. 6	0.630	5900	10.2	1.07	1.34	62.7
No. 7	0.600	5960	10.1	1.01	1.16	62.4

[a] Calculated from equation; surface area = $\left[\frac{980\,(d_c - d_f)}{knK}\right]^{1/2}$.

[b] Water in excess of w_i at sand-carrying capacity proportion.

2 being the highest. The other limes, Nos. 1, 3, and 5, fall more or less in a group. Figure 6 (*b*) shows that there is a rough relation between the immobile-water factors and flow after suction as would be expected. Plotting the values for w_i against plasticity shows no relation between these two factors. Plotting w_i against sand-carrying capacity also shows no exact relation although the limes having the largest immobile-water factors do have the higher sand-carrying capacity.

The apparent specific surface and the average apparent particle size have been calculated from the sedimentation data. These values are given in Table IV. It is not known how close these values are to the true surface and size values, but it is believed that they are not absolute values because of the fact that complete dispersion was not obtained in the pastes, as used, and agglomeration or flocculation would be reflected in the results recorded. In other words the true value for specific surface would be larger if complete dispersion of the particles was obtained. For this reason the true average particle size would be smaller than shown. In any event, the values given are valuable for purposes of comparison. The thickness of the immobile-water layer has been calculated from these data and is recorded in the fifth column of Table IV. These water films are roughly of the order of magnitude of one tenth the average particle diameter or approximately 1μ in thickness.

The difference between the mixing water in milliliters per gram of lime and that held immobile (w_i in milliliters per gram of lime) is given in the sixth column. The mixing water is based on that required to plasticize the mortar at the proportion of sand-carrying capacity. The amount of water in excess of the immobile-water varies with the lime, but with the exception of lime No. 2, the percentage of the total water required is practically constant, as shown in the seventh column. Approximately 38 to 40 per cent of the total mixing water required at this proportion of sand and lime is immobile water. The assumption is made here that the same sort of action occurs in the mortar as in the paste. It is intended to make a more comprehensive investigation along these lines in order that sufficient data will be obtained to make definite conclusions warranted.

SUMMARY

Hydrated limes may be roughly classified as regards their plasticizing ability in a mortar by an examination of the mixing-water requirements for mortars of variable sand content and fixed consistency. Six of the seven limes investigated could be classified on the premise that the higher the mixing-water requirement, the higher the plasticizing ability.

Relative sand-carrying capacities of the lime hydrates were measured most accurately with the extrusion device. The void method presented checked the extrusion device most consistently.

No apparent relation was found between the plasticity figure and flow after suction or the plasticity figure and sand-carrying capacity.

A rough relationship was shown between flow after suction and sand-carrying capacity.

Hydrated limes may be roughly classified as to their plasticizing ability in a mortar by sedimentation tests and the determination of the immobile-water factor.

A rough relationship is shown between the immobile-water factor and flow after suction. No apparent relationship is shown between the immobile-water factor and plasticity.

DISCUSSION

Mr. G. J. Fink.[1]—I should like to ask Mr. Staley whether I am correct in the inference that he uses the term particles to refer to the aggregates that exist in the water dispersions.

Mr. Howard R. Staley.[2]—That is correct. The term particle has been used to designate the unit which acts as a single particle whether it be a single particle or a group of particles. If each single particle acted as a unit, then I believe the surface areas determined by this method would be much larger than those given. In this instance, we are concerned with the effective rather than the true surface area.

Mr. F. O. Anderegg.[3]—Mr. Staley has made a contribution to a poorly developed science, which underlies the technology of water suspensions of lime involved in mortars and plasters. I have had similar experience with lack of correlation between plasticity values obtained with neat putties on the Emley plasticimeter and the workabilities or sand-carrying capacities of limes. The best matrix I have encountered carried 4.5 volumes of Newark, Ohio, sand, but gave a plasticity value below 150. The changes in rheological properties in going from a neat putty to a sanded mortar are so great that it is not surprising to find differences. In a mortar the ratio of highest to lowest water content for working consistency among limes may reach 150 per cent, as compared with a ratio of 230 per cent for similar putties at normal consistency.

The extrusion apparatus has failed to correlate in our experiments with the masons' requirements. The reason for this may lie in our sands, which run coarser than Mr. Staley's. The method of mixing may be a factor, since we mix for 10 min. mechanically before running the extrusion experiment. We have had to reduce the diameter of the outlet from the extrusion apparatus from 1.5 to 0.9 in. in order to satisfy the masons' needs. This is a reduction of 64 per cent. I should like to ask Mr. Staley whether he has experimented with masonry sand from the Ohio or other midwestern rivers in the extrusion apparatus.

Mr. Staley.—The coarsest sand used had a fineness modulus of 2.25 while the values reported were obtained with a sand of fineness modulus of 2.15. Using a sand of 2.25 fineness modulus did not alter the results. No work has been done with the river sands of the Mid-West.

The procedure used by Mr. Anderegg for mixing the mortars would affect the results of the sand-carrying capacity test as a longer mixing period, or reworking after some long period of rest, would yield a higher value for this factor. The procedure given must be followed to obtain the same results.

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THE EFFECT OF FINE AGGREGATE ON THE DURABILITY OF MORTARS[1]

BY C. A. HUGHES[2] AND KENNETH A. ANDERSEN[3]

SYNOPSIS

This paper presents a comparison of data from laboratory durability tests of fine aggregates and mortars made therefrom and data from natural weathering of the mortars.

Eighteen fine aggregates were subjected to sodium and magnesium sulfate tests and to an accelerated freezing-and-thawing test. A fast and a slow laboratory freezing-and thawing test was used for the mortar specimens, which were also placed outside in 1 in. of water. The investigation showed no usable relation between the data from the laboratory durability tests on the mortars and the data on companion specimens after 8 yr. of natural weathering. The compressive strength is shown to be unreliable as a criterion of the durability of mortars made from different fine aggregates. An interpretation of the magnesium sulfate test is presented, which, if accepted, leads to the adoption of a lower limit as well as an upper limit to the magnesium sulfate soundness test. The size and shape of the test specimen and the criterion used in judging resistance to either laboratory cycles or natural weathering are shown to be important factors in rating for durability.

The durability herein considered is the resistance to outside exposure involving moisture and temperature changes. More specifically, the exposure considered is that existing at the ground line or the water line in concrete structures erected in northern climates.

In an attempt to guard against the use of materials, proportions, and methods of curing that would cause short life in the field, many laboratory control tests on the aggregate, mortar, or the concrete have been proposed and, in certain cases, applied. All such tests attempt to predict behavior in service from data obtained in a short time in the laboratory. The desirability of such control tests is obvious. However, consideration of the disregard of the time element and the excess of factors affecting the durability in the field over the factors included in the laboratory tests have led to a healthy scepticism as to the correlation of laboratory and field results. Consequently the number of tests adopted by responsible organizations for control of durability are relatively few compared to the number that have been proposed.

Because concrete of even very poor quality requires four, five, or more years of service to develop evident symptoms of distress, progress in developing a laboratory test having definite correlation with field experience has been, and probably will remain, very slow.

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[2] Associate Professor of Structural Engineering, University of Minnesota, Minneapolis, Minn.

[3] Research Assistant, Engineering Experiment Station, University of Minnesota, Minneapolis, Minn.

This investigation was originated to study the relations between data from the sodium and magnesium sulfate tests on fine aggregates and data from various laboratory tests on mortars made from the fine aggregates. Because mortar specimens were exposed also to natural weathering, the investigation has served to supply data on the correlation of data from accelerated laboratory tests and data from natural weathering.

TABLE I.—PROPERTIES OF FINE AGGREGATES.

Data are averages for two tests.

Identification	Source	Durability Rating According to Sender	Colorimetric Test Plate Number	Clay and Silt, per cent by weight	Bulk Specific Gravity	Absorption, per cent by weight	Shale by Flotation, per cent by weight	Fineness Modulus	Surface Modulus	Sodium Sulfate at 70 F., weighted average loss after 5 cycles	Magnesium Sulfate, weighted average loss after 5 cycles	Cycle *G*, Freezing and Thawing, weighted average loss after 30 cycles
No. 1.....	Irondequoit, N. Y.	No. 3 of 23 sands	I	1.3	2.66	0.82	0	2.87	21.5	4.0	13.1	4.4
No. 2.....	Jamestown, N. Y.	No. 10 of 23 sands	I	2.5	2.60	1.73	0	2.92	21.8	4.2	24.9	8.4
No. 3.....	Springville, N. Y.	......	I–II	1.8	2.58	1.77	0	2.94	20.8	6.8	25.4	8.3
No. 4.....	Syracuse, N. Y.	No. 19 of 23 sands	I	4.2	2.58	2.30	0	2.90	20.0	16.5	36.6	14.1
No. 5.....	Buffalo, N. Y.	Border-Line	I	0.7	2.67	1.27	0	2.76	20.4	3.7	19.0	7.3
No. 6.....	Buffalo, N. Y.	Satisfactory	I–II	0.9	2.64	0.90	0	3.31	21.6	3.2	12.3	5.5
No. 7.....	Boonville, N. Y.	Satisfactory	I	0.3	2.65	0.27	0	2.84	19.8	2.0	5.6	2.4
No. 8.....	Sterling Creek, N. Y.	Unsatisfactory	I	1.8	2.59	2.10	0	3.15	18.5	13.2	32.2	12.2
No. 9.....	Pennsylvania	Good	I	1.0	2.60	1.16	0	2.77	22.9	3.9	10.2	4.7
No. 10....	Crosby, Minn.	Poor	II	2.8	2.42	4.30	3.9	3.22	18.8	17.4	...	11.8
No. 11....	Moose Lake, Minn.	......	I–II	3.4	2.68	1.31	0	3.38	12.8	3.5	12.0	3.1
No. 12....	Appleton, Minn.	......	I–II	0.6	2.58	1.90	1.8	2.74	23.1	6.7	11.9	6.4
No. 13....	Trap screenings with dust	......	I	8.5	2.87	1.09	0	3.19	24.3	...	...	...
No. 14....	Trap screenings without dust	......	I	0.3	2.94	0.44	0	3.78	11.7	2.6	7.3	1.4
No. 15....	Minneapolis, Minn.	Good	I	0.4	2.68	0.63	...	3.19	15.9	3.6	8.2	2.2
No. 16....	Haydite	......	I	...	1.68	6.79	...	3.56	14.6	3.0	44.2	1.4
No. 17....	Limestone screenings with dust	......	I	22.8	2.49	3.34	...	2.50	38.0	...	...	...
No. 18....	Limestone screenings without dust	......	I	7.3	2.56	2.19	...	3.25	20.4	40.2	47.4	41.3

FINE AGGREGATES

The fine aggregates were received from various sources in response to a request for fine aggregates that had been rated as (*a*) durable, (*b*) nondurable, and (*c*) of intermediate quality.

The properties of the aggregates, their sources, and the durability ratings given by the senders are shown in Table I. Of the 18 fine aggregates, 13 were natural fine aggregates, 4 were crushed material, and 1 was an artificial fine aggregate.

Test Procedures.—The procedures of this Society pertaining in 1931 to the colorimetric test, the determination of the percentage of clay and silt, and the mechanical analysis were closely followed. The percentage of shale was determined as the fraction separated by a liquid having a specific gravity of 1.98 according to the A.A.S.H.O. method. The specific gravities were determined by use of Dunagan's apparatus and the absorption by the difference in weight between saturated surface-dry and oven-dry samples of approximately 1000 g.

In addition to the sodium and magnesium sulfate tests on the fine aggregate, an accelerated freezing-and-thawing test was used that had previously been developed in this laboratory for use with coarse aggregate.[4] A similar method has recently been described by Dawley.[5]

The sodium sulfate test was carried out as recommended by the A.S.T.M. Tentative Method of Test for Soundness of Fine Aggregate by Use of Sodium Sulfate (C 88 – 31 T).[6] The magnesium

TABLE II.—PROPERTIES OF MORTARS OTHER THAN DURABILITY.

Fine Aggregate	Water-Cement Ratio, gal. per sack	Paste Ratio, $v + c$[a]	Aggregate-Cement Ratio, a/c	Voids-Cement Ratio, v/c	Air Voids	Cement Factor, sacks per cu. yd.	Compressive Strength of 2-in. Cubes, psi.	
							Age, 7 days	Age, 28 days
No. 1	5.07	0.463	3.02	1.60	0.04	10.0	5300	7470
	6.76	0.418	4.70	2.37	0.06	7.0	2740	4740
No. 2	5.07	0.474	2.83	1.55	0.03	10.4	5510	7535
	6.76	0.430	4.23	2.18	0.04	7.6	2690	4560
No. 3	5.07	0.470	2.86	1.54	0.03	10.3	4780	6820
	6.76	0.418	4.45	2.19	0.04	7.3	2470	4350
No. 4	5.07	0.507	2.42	1.49	0.02	11.4	4700	6040
	6.76	0.449	3.85	2.14	0.04	8.0	2465	4100
No. 5	5.07	0.474	2.91	1.62	0.04	10.1	5635	7615
	6.76	0.429	4.46	2.35	0.06	7.2	2970	4970
No. 6	5.07	0.389	4.07	1.59	0.03	8.4	5340	7230
	6.76	0.367	5.45	2.16	0.04	6.5	2925	4850
No. 7	5.07	0.450	3.22	1.63	0.04	9.6	4940	6910
	6.76	0.424	4.37	2.21	0.05	7.4	2655	4630
No. 8	5.07	0.462	2.92	1.51	0.02	10.3	4925	7050
	6.76	0.419	4.27	2.08	0.03	7.6	3095	4610
No. 9	5.07	0.467	2.96	1.59	0.04	10.1	5075	7690
	6.76	0.427	4.41	2.28	0.06	7.3	2550	4490
No. 10	5.07	0.455	2.88	1.41	0	10.6	4155	6270
	6.76	0.408	4.43	1.97	0.02	7.6	2420	4020
No. 11	5.07	0.444	3.14	1.51	0.02	9.9	4625	6650
	6.76	0.404	4.65	2.16	0.04	7.2	2125	3780
No. 12	5.07	0.462	3.04	1.61	0.04	9.9	4575	6080
	6.76	0.424	4.47	2.29	0.06	7.2	2595	3385
No. 13	5.07	0.523	2.22	1.43	0.01	12.1	6635	8630
	6.76	0.471	3.30	1.93	0.01	9.0	3920	5940
No. 14	5.07	0.515	2.29	1.43	0.01	11.9	6740	8940
	6.76	0.489	3.15	2.02	0.02	9.1	2925	5360
No. 15	5.07	0.428	3.42	1.56	0.03	9.4	4640	6598
	6.76	0.407	4.66	2.20	0.04	7.1	2350	4600
No. 16	5.07	0.635	1.48	1.57	0.04	13.9	6740	6042
	6.76	0.600	2.15	2.22	0.07	10.4	4140	5610
No. 17	5.07	0.567	1.84	1.41	0	13.2	5671	6638
	6.76	0.426	3.98	1.96	0.01	8.1	3110	4840
No. 18	5.07	0.506	2.33	1.39	0	11.9	6071	7145
	6.76	0.443	3.57	1.84	0	8.8	3610	5035

[a] Ratio of the absolute volume of cement paste, water plus air voids plus cement, to the volume of mortar.

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[4] F. C. Lang and C. A. Hughes, "Accelerated Freezing and Thawing as a Quality Test for Concrete Aggregates," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 435 (1931).

[5] E. R. Dawley, "Correlation Between Rapid Immersion and Standard Four Cycle per Day Freezing-and-Thawing Tests," *Journal*, Am. Concrete Inst., Vol. 12, No. 2, November, 1940, p. 157.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 773 (1931); also 1931 Book of A.S.T.M. Tentative Standards, p. 267.

sulfate tests were made according to the same procedure with the exception that a saturated magnesium sulfate solution was used in place of the sodium sulfate solution.

The accelerated freezing-and-thawing cycle, hereafter referred to as cycle *G*, consisted of 5 min. in water at 200 to 212 F., 5 min. in water at 35 to 40 F., 30 min. in calcium chloride brine at 0 ± 10 F., and 5 min. draining in air at 70 ± 5 F. Thus, 45 min. were required per cycle. Ten cycles were run per day. The aggregate samples were prepared according to the procedure used in the sulfate tests. Each aggregate fraction was contained in a small metal pan with a perforated lid and a bottom of 100-mesh screen.

Criterions for Durability Tests.—The criterion used for all three durability tests is the weighted percentage loss in weight calculated according to the method described in Method C 88 – 31 T.[6]

MORTARS

Proportions:

In proportioning mortars from fine aggregates differing in shape, surface characteristics, and grading, either the water-cement ratio, the consistency, or the paste ratio must vary. Because previous data indicated the water-cement ratio to be a more important factor than the paste ratio a uniform consistency was known to minimize irregularities in molding, the water-cement ratio and the consistency were held constant. Two water-cement ratios, 0.45 and 0.60 by weight, or 5.07 and 6.76 gal. per sack were used. The consistency was kept constant at a flow of 60 ± 5 using thirty $\frac{1}{8}$-in. drops of a 10-in. flow table. The quantity of aggregate for each fine aggregate was determined by trial. Certain properties of these mixes are given in Table II.

Specimens:

The specimens were 2-in. cubes or 2 by 2 by 10-in. beams cast in metal molds. Brass plugs were cast in the ends of the beams for use in measuring changes in length.

Durability Cycle:

Cycle P.—The specimens were alternately submerged under water at 70 F. and placed in air at 0 ± 10 F.

Cycle H.—The specimens were alternately submerged in water at 70 F. and under 150 psi. pressure and placed in air at 0 ± 10 F.

In cycles *P* and *H* changes from one condition to the other were made at 8 a.m., noon, and 5 p.m. daily except Sunday. Hence there were $1\frac{1}{2}$ cycles per day and 9 cycles per week

Sodium Sulfate Exposure.—The specimens were placed in a 10 per cent aqueous solution of sodium sulfate. This solution was renewed once per month.

Natural Weathering Exposure.—Except for a few beams stored outside on a mesh rack, the natural weathering specimens, both beams and cubes, were placed in pans containing water to a depth of 1 in.

Curing:

The curing was not the same for all specimens. The beams in cycle *H* were started in freezing and thawing after 28 days of submersion in water at 70 F. The natural weathering beams were placed outside on February 13, 1934, after 42 days of submersion in water. The cubes in cycles *P* and *H*, for natural weathering, and for sodium sulfate exposure were cured for $6\frac{1}{2}$ months under water. The natural weathering cubes were placed outside on October 13, 1933.

TABLE III.—PROPERTIES OF MORTARS—2-IN. CUBES.

Data shown are the averages for three specimens.

Fine Aggregate	Laboratory Tests								Natural Weathering					
	Number of *H* Cycles at				Number of *P* Cycles at		Number of Days of Immersion in 10 per cent Na_2SO_4 Solution		Change in Weight After 8 yr., per cent		Number of Years at			
	10 per cent Loss in Weight		30 per cent Loss in Weight		10 per cent Loss in Weight	30 per cent Loss in Weight	30 per cent Loss in Weight	Rating 4			10 per cent Loss in Weight		30 per cent Loss in Weight	
	w/c = 5.07	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76	*w/c* = 6.76	*w/c* = 6.76	*w/c* = 6.76	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76
No. 1	64	31	75	40	204	22.6%[a]	106	75	−1.5	−5.7	...	...	...	...
No. 2	76	35	84	48	116	571	107	68	−1.5	−3.7	...	...	...	...
No. 3	67	34	79	36	106	405	79	62	−1.9	−8.8	...	...	...	...
No. 4	52	33	62	35	132	318	84	62	−3.0	−9.4	...	...	...	...
No. 5	85	39	104	54	170	28.8%[a]	87	79	−1.7	−3.8	...	...	...	...
No. 6	76	36	93	49	248	573	77	62	−4.9	−12.4	...	6¾	...	...
No. 7	76	36	104	51	113	299	67	62	−5.2	−74.0	...	4¾	...	6¾
No. 8	59	31	72	40	186	528	79	62	−16.1	−20.9	7½	6	...	...
No. 9	111	36	138	53	162	434	81	62	−1.7	−20.0	...	5¾	...	...
No. 10	44	18	48	25	58	138	89	62	−38.6	7 yr.[c]	7	2¾	7¾	4¼
No. 11	78	31	103	55	108	367	69	62	−4.7	−31.6	...	3¾	...	7¾
No. 12	61	28	76	43	112	448	95	75	−4.2	−11.4	...	7¾	...	...
No. 13	153	58	175	69	160	317	174	82	−2.7	−24.5	...	7¼	...	...
No. 14	217	70	271	108	161	367	153	79	−4.0	−58.8	...	5½	...	6¾
No. 15	77	34	95	47	128	479	80	62	−3.0	−30.6	...	7	...	8
No. 16	43	23	60	44	9%[a]	9.0%[a]	−11.8%[b]	106	−27.8	−8.9	7	...	...	...
No. 17	43	15	47	18	30	41	+6.0%[b]	>183	−1.2	−11.2	...	7½	...	...
No. 18	58	17	62	24	50	91	266	148	−1.8	−9.0	...	...	...	...

[a] Percentage loss in weight at 651 *P* Cycles.

[b] Percentage change in weight after 482 days immersion in 10 per cent Na_2SO_4 solution.

[c] Number of years for 100 per cent disintegration.

TABLE IV.—PROPERTIES OF MORTARS—2 BY 2 BY 10-IN. BEAMS.

Data are the averages for 2 specimens except as noted.

Fine Aggregate	Laboratory Tests				Natural Weathering							
	Number of *H* Cycles				Relative Expansion After 8 yr. × 10^6			Specimens in 1 in. of Water, yr.		Change in Weight After 8 yr., per cent		
	Relative Expansion of 0.0005		Relative Expansion of 0.003		Specimens in 1 in. of Water		Specimens on a Wire Rack	Relative Expansion of 0.0005		Specimens in 1 in. of Water		Specimens on a Wire Rack
	w/c = 5.07	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76	*w/c* = 5.07	*w/c* = 6.76	*w/c* = 6.76
No. 1	24	16	68	48	+806	+315	−402	6	...	−0.6	−0.5	−4.2
No. 2	21	17[a]	70	53[a]	+51	+15	...	...	...	−0.6	−1.4	...
No. 3	18	13[a]	54	40[a]	+175	+149	...	...	...	−0.8	−1.8	...
No. 4	8	6[a]	43	25[a]	−1	+159	−933	...	...	−1.2	−3.9	−3.9
No. 5	25	20	70	55	+384	+185	...	...	...	−0.6	−0.9	...
No. 6	10	12[a]	46[a]	42	+476	+316	...	...	...	−0.8	−0.9	...
No. 7	25	12	60	42	+1370	+1289	...	4¼	4½	−0.6	−0.6	...
No. 8	8	6	24	24	+456	+323	...	...	...	−0.9	−1.1	...
No. 9	...	...	...	...	...	...	...	...	...	...	...	...
No. 10	...	...	...	...	...	...	...	...	...	...	...	...
No. 11	...	...	...	...	...	...	...	...	...	...	...	...
No. 12	22	12	64	38	+3226	+616	...	4½	4¾	+0.4	−0.6	...
No. 13	17	12	50	44	+93	+266	−443	...	...	−0.6	−7.8	−3.1
No. 14	19	22	60	64	+647	+327	−324	7¼	...	−1.2	−3.4	−4.0
No. 15	22	17	54	54	+2175	+882	+38	4½	5	−0.2	−1.2	−3.2
No. 16	12	16	27	...	+2324[b]	+739	−268	3½	6	[e]	−3.4	−6.3
No. 17	2	1	13	4	+1911[c]	+3631[d]	−694	3½	1¾	−93.0	5 yr.[f]	−3.2
No. 18	5	2	18	10	+436	+89	...	...	...	−1.45	−1.2	...

[a] Average of 4 specimens.

[b] Expansion for one specimen at 7 yr. The companion specimen was in 2 pieces the sixth year.

[c] Expansion at 6 yr.

[d] Expansion at 4 yr.

[e] One specimen was in 3 pieces in eighth year and the other in 2 pieces the sixth year.

[f] Number of years for 100 per cent disintegration.

Criterions of Durability:

Percentage Loss in Weight.—The specimens from the laboratory cycles and natural weathering were blown off with a standardized air stream and weighed periodically. From these data the numbers of cycles or the number of years of exposure to natural weathering causing a specified percentage loss in weight were calculated. The natural weathering specimens were submerged for 48 to 72 hr. prior to blowing off and weighing. At the time these tests were run, 30 per cent loss in weight was considered an adequate end point. The authors have since been convinced that the use of a higher percentage loss in weight, say 50 per cent, is preferable. Because very few specimens subjected to natural weathering had reached 30 per cent loss in weight at the time of writing, 10 per cent loss in weight was used as a temporary end point for these specimens. When comparing natural weathering and laboratory cycles, a 10 per cent end point was used for the laboratory cycles.

Expansion.—The changes in length of the 2 by 2 by 10-in. beams were measured by a specially mounted Ames dial reading, by interpolation, to 0.00001 in., and thus, for a 10-in. specimen, giving relative deformation to 0.000001. Duplicate readings indicate the reading error, expressed as a relative deformation, is less than 10 points, or 0.00001. All readings were taken with the beams submerged in water at 70 F. An end point at a relative expansion of 0.003 was used for the laboratory specimens. A higher expansion is preferred but for some specimens disintegration around the ends prohibits the use of higher values. Because the relative expansions of the majority of the natural weathering beams had not yet reached 0.003 at the time of writing, a relative expansion of 0.0005 was used as a temporary end point. The number of years of natural weathering as given is more accurately the number of winters of exposure. For the beams placed outside on February 13, 1934, a full year was counted for the exposure of the remainder of the winter.

Ratings for Mortars Exposed to 10 per cent Sodium Sulfate Solution.—The cubes were rated periodically by visual inspection from 0 for no evidence of disintegration to rating 4 for one corner rounded. Rating 4 was considered an end point for visual ratings.

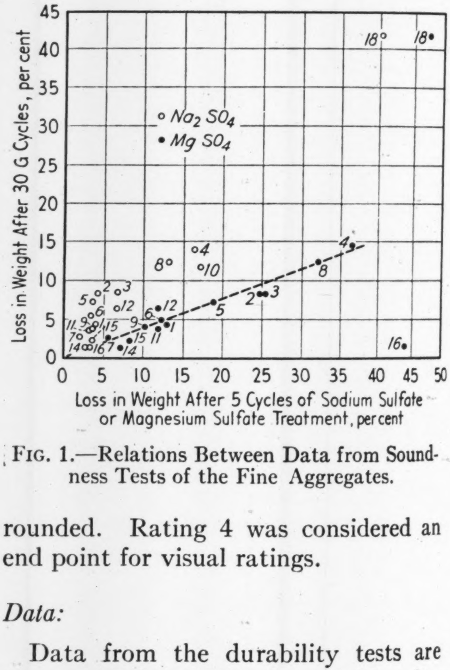

FIG. 1.—Relations Between Data from Soundness Tests of the Fine Aggregates.

Data:

Data from the durability tests are given in Table III for the 2-in. cubes and in Table IV for the 2 by 2 by 10-in. beams. Because some of the natural weathering specimens had not reached the defined end point, the percentage loss in weight and change in length after 8 yr. of natural weathering are given in addition to the number of years required to reach the end point.

In Fig. 1, the data from the magnesium and sodium sulfate tests on the fine aggregates are plotted against the corresponding data for cycle *G*. Except for fine aggregates Nos. 16 and 18—haydite and crushed limestone, respectively—a definite relation is shown between the data for cycle *G*, and the magnesium sulfate test. A trend is indicated by the sodium sulfate data. Hence, the same properties of the natural aggregate appears to be measured by the three cycles. Because of the greater range in the data from the magnesium sulfate test, it should be given preference. It should be noted that the haydite aggregate suffered little from frost action or from sodium sulfate but was markedly disintegrated by the magnesium sulfate. Damage to the limestone was severe in all three cycles but was relatively worse in cycle *G*.

If the accelerated durability tests on the aggregate are significant, definite relations should be obtained between the data therefrom and durability data obtained on mortars made from the fine aggregate. Data for the aggregates are plotted against the data for the mortars for both the laboratory cycles and for natural weathering in Fig. 2. From sections *a*, *b*, *g*, *h*, *m*, and *n*, it will be seen that fairly definite trends exist for the natural aggregates except in section *a*. Consequently the soundness tests on the natural aggregates are indicative of the resistance of mortars made from them when subjected to the freezing-and-thawing exposure of cycle *H*. Whether expansion of beams or loss in weight of cubes is used, the limestone mortars, No. 18, are definitely low in resistance to cycle *H*. The trap aggregate mortars are definitely high in resistance when the percentage loss in weight is considered, which is in line with the general impression as to the durability of concretes made with trap aggregate.

Proceeding to sections *d*, *j*, and *p* of Fig. 2, it is evident that the resistance of mortar to 10 per cent sodium sulfate solution is independent of the quality of natural aggregates as measured by the sulfate tests or cycle *G*. An interesting feature of these panels, however, is the high resistance to sodium sulfate of mortars made from limestone, No. 18, or from haydite, No. 16. It will be seen from Table III that the mortar made from aggregate No. 17, limestone screenings with dust, showed an increase in weight of 6 per cent after 482 days immersion in 10 per cent sodium sulfate. The high resistance of the haydite specimens can be explained on the basis of pozzuolanic action of the haydite dust demonstrated by another investigation of this laboratory.[7] No explanation is available for the high resistance of the mortars made from limestone screenings.

In sections *c* and *o* of Fig. 2, no relation is apparent between the sodium sulfate and cycle *G* data and the resistance of the mortars to the slow cycle *P*. In section *i*, however, trends are shown which indicate that maximum resistance to cycle *P* is obtained if the weighted average loss in the magnesium sulfate test is in the neighborhood of 18 per cent. The conception of an optimum percentage loss in the magnesium sulfate test is unorthodox. However, study of the data for natural weathering in Figs. 2 and 3 will show that it is probably correct. It is at once evident from sections *e*, *f*, *k*, *l*, *q*, and *r* that the use of an aggregate having a low loss in magnesium sulfate is no assurance that the resistance of the mortar to natural weathering will be high. On the contrary, higher weathering resistance may be obtained

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[7] C. A. Hughes and A. S. Levens, "Strength and Shrinkage of Mortars Made with Blends of Portland Cement and Pozzuolanic Materials," *Proceedings*, Am. Concrete Inst., Vol. 27, p. 317.

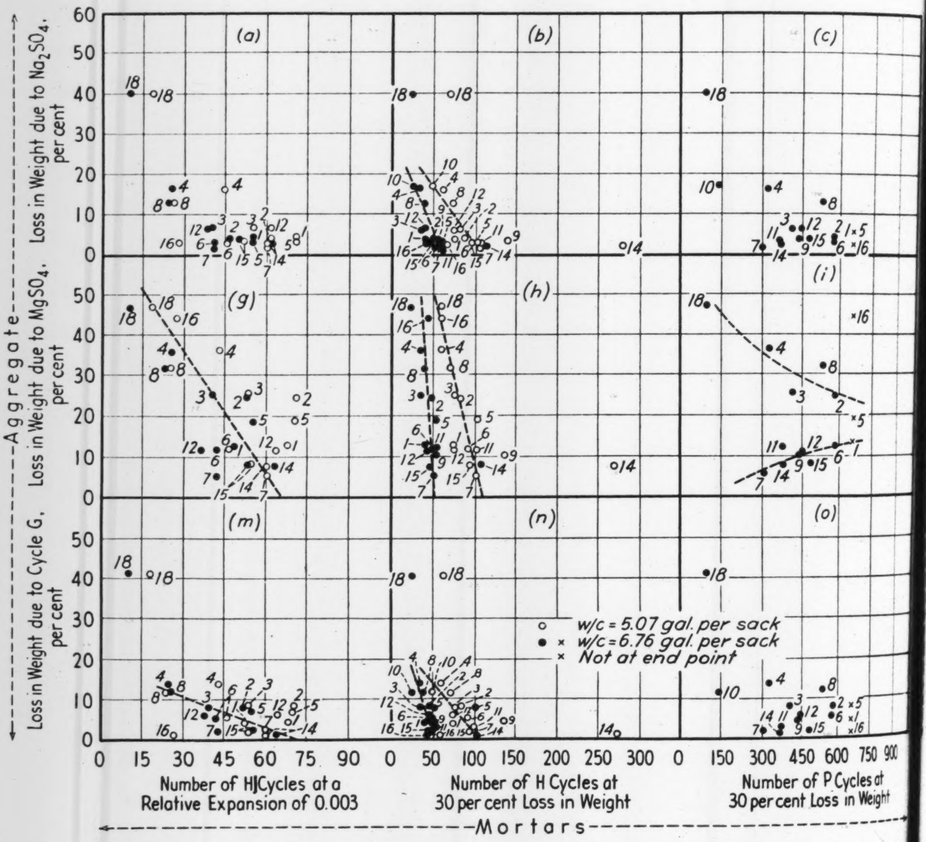
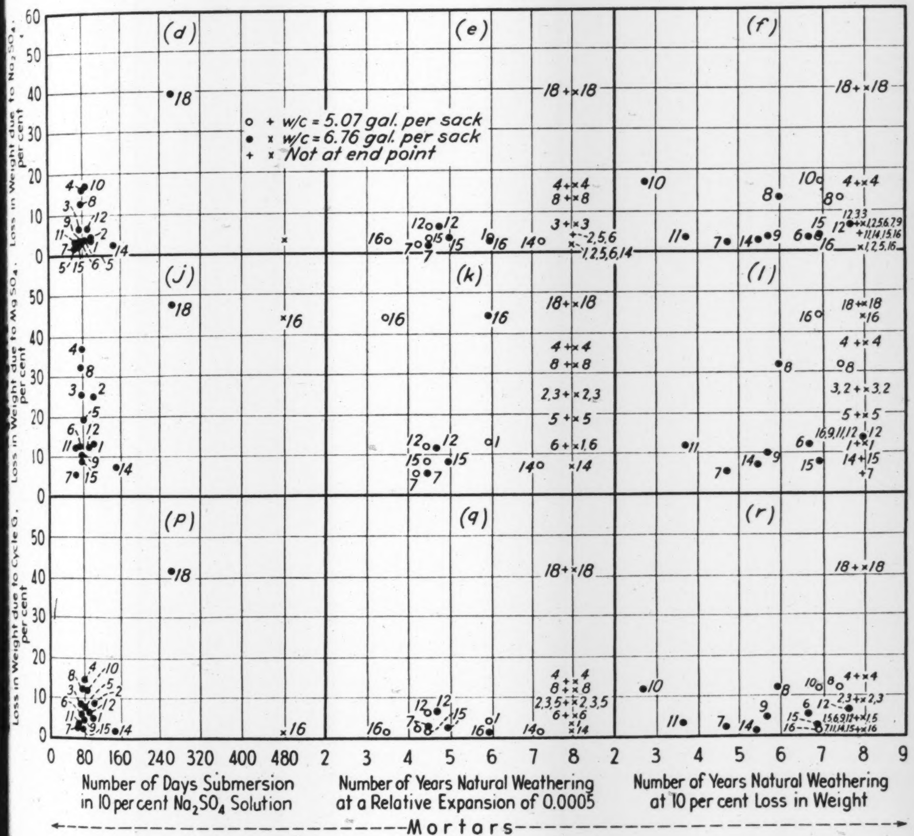


FIG. 2.—Relations Between Data from Soundness Tests of the Fi gregate

All plotted points are averages for from two to four specime The subs



the Fi gregates and Data from Durability Tests on Mortars Made Therefrom.
r specime The subscripts are the aggregate identification numbers.

from mortars made from aggregates having higher losses in magnesium sulfate. Considering only those mortars which did not reach an end point at 8 yr. natural weathering for both water-cement ratios, we find Nos. 2, 3, and 5 at about the optimum loss in magnesium sulfate and for higher or lower magnesium sulfate losses, Nos. 18, 4, 8, and 6 for the beams (section *k*) and 18, 4, and 1 for the cubes (section *l*). From the data of Table III it can be found that for aggregates Nos. 2, 3, and 5 the average expansion at 8 yr. is 0.000160 and the average loss in weight is 3.6 per cent, whereas for the specimens mentioned above made from aggregates having higher or lower losses in the magnesium sulfate test the average expansion is 0.000282 and the average loss in weight is 5.1 per cent. Hence, in all probability, specimens Nos. 2, 3, and 5 will prove to be more durable than the remaining specimens which have not reached an end point. If this is true, then when the data are complete for sections *l* and *k*, trends similar to those indicated in section *i* will be shown.

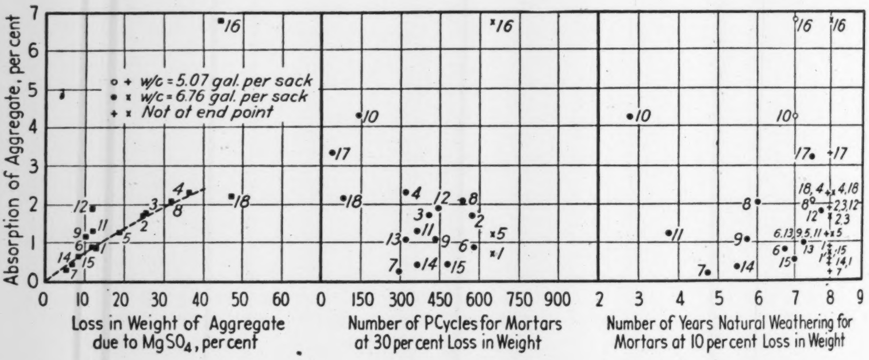


FIG. 3.—The Effect of the Absorption of the Fine Aggregate on Magnesium Sulfate Soundness Loss and the Resistance of Mortars to Cycle *P* and to Natural Weathering.

It is known by those engaged in durability studies that the use of smooth-surfaced aggregates having low absorptions will result in mortars low in resistance to freezing and thawing because of failure in bond between the cement paste and the aggregate. This action is very marked when high-consistency or high water-cement ratio pastes are used. Examination of disintegrated specimens shows the aggregate to be almost entirely free from paste. The data shown below were obtained on 2-in. cubes submitted to this laboratory. Aggregate A appeared to be 100 per cent siliceous and had exceptionally smooth surfaced and rounded particles. Aggregate B contained considerable limestone. Unfortunately, aggregate samples were not received and consequently the absorptions are not known. However, there is little question that aggregate B was considerably more absorptive than aggregate A.

Water-Cement Ratio, gal. per sack	Number of *P* Cycles at 15 per cent Loss in Weight		Ratio of B to A
	Aggregate A	Aggregate B	
7.56	34	201	6
5.52	190	372	2

The decrease in resistance to the freezing and thawing caused by the use of aggregate A is most marked for the high water-cement ratio. Hence, it seems evident that the optimum absorption or the optimum magnesium sulfate loss will vary with the consistency of the paste to be used with the aggregate.

For brick masonry it has been shown that the maximum bond is obtained for an optimum suction rate which varies with the consistency of the mortar.[8] It seems likely that some such line of thought could be applied to mortars and concrete with profit. The fine aggregates of the investigation of this paper were saturated surface dry when mixed. Had they been dry when mixed it is possible that the durability of the aggregates of low absorption might have been improved.

In Fig. 3, the absorptions of the aggregates are plotted against the data from the magnesium sulfate soundness test, from cycle *P*, and from natural weathering. A good relation was obtained between the magnesium sulfate loss and the absorption of the aggregate, though No. 16 lies far away from the trend shown and it is probable that No. 10 would also if the magnesium sulfate loss were known. It will be seen on examination that the cycle *P* and natural weathering data indicate the mortars having highest resistance were made from aggregate having absorptions somewhat above 1 per cent. Comparison of Figs. 2 and 3 will show that the use of an optimum loss in the magnesium sulfate soundness test is a more efficient method of selecting a fine aggregate for mortar durability than is the use of an optimum percentage absorption of the aggregate.

Failure of specimens made from aggregate low in absorption has been discussed. Failure of specimens made with aggregates high in absorption is thought to be due to differential movements resulting from the expansion of the absorbed water on freezing as well as to the softness of the aggregates that usually accompanies high absorption.

Again referring to Fig. 2, it will be seen that the mortar made from trap screenings without dust, No. 14, is exceptionally high in resistance to the fast cycle *H* when the percentage loss in weight from a cube is used as the criterion, and is among the specimens of highest resistance when the expansion of beams is used as a criterion. In the slow cycle *P* and in natural weathering the resistance of the trap mortars is relatively much less. For example, in natural weathering, using the loss in weight as a criterion the trap mortar is fourth lowest in resistance, failure occurring in bond. The mortars made from aggregate No. 16, haydite, having nearly 7 per cent absorption had very low resistance in the fast cycle *H*, and relatively high resistance in the slow cycle *P* and in natural weathering. The behavior of the specimens made from limestone aggregate No. 18 differs radically in the laboratory cycles and in natural weathering. Concrete roads using limestone No. 18 have given excellent service, whereas all laboratory tests on this material have indicated it to be very low in resistance to freezing and thawing. On the other hand, the mortar beams made from the limestone screenings with dust showed rapid disintegration in natural weathering (see Table IV) reaching the end point for expansion in $3\frac{1}{2}$ and $1\frac{3}{4}$ yr. for the low and high water-cement ratios, respectively. The losses in weight for the beams were 93 per cent at 8 yr. and 100 per cent at 5 yr. for the low and high water-cement ratios, whereas the losses for the corresponding cubes were 1.2 and 11.2 per cent. No explanation

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[8] L. A. Palmer and D. A. Parson, "A Study of the Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper 683*, Nat. Bureau Standards, Figs. 12 and 13, p. 633.

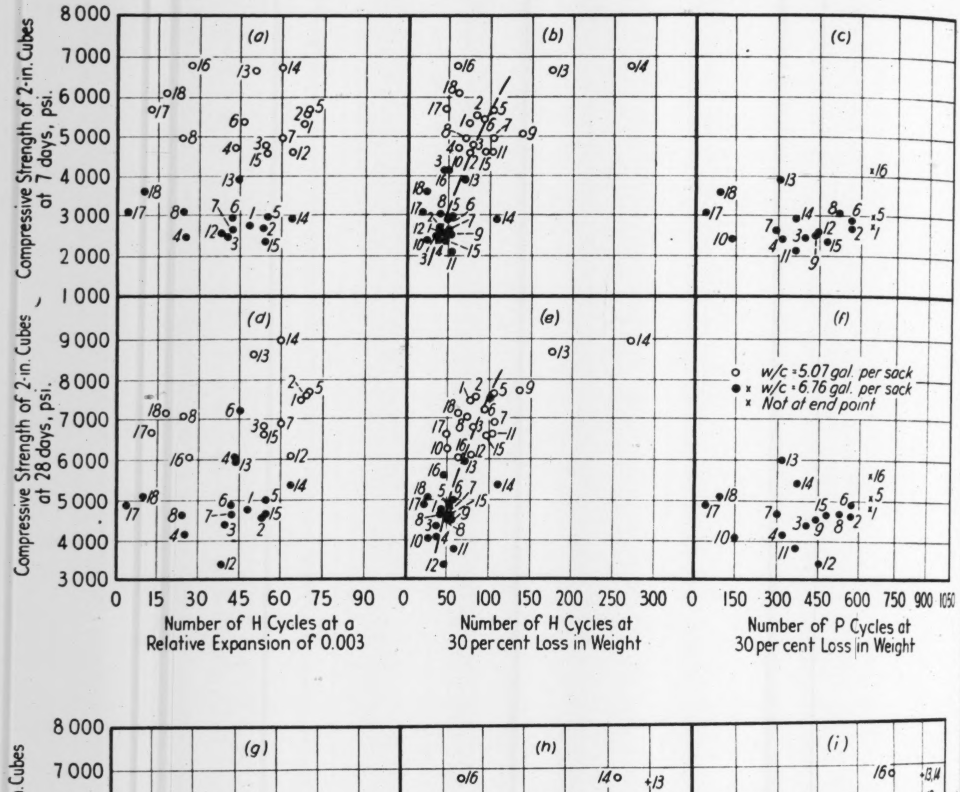

FIG. 4.—The Compressive Strength as a Criterion of Durability.

is known for the marked discrepancy in behavior of beams and cubes made from the same mix. The much higher percentage of dust in aggregate No. 17 as compared with aggregate No. 18 probably explains the greater durability of the mortars made from the latter aggregate.

Because time is required for disintegration in service and time is known to cause modifications in the stress-strain relations of the cement paste, it is believed that the longer the laboratory cycle, the greater are its chances to correlate with natural weathering. The data of Fig. 2 indicate cycle *H* to be definitely inferior to cycle *P* as a means of predicting the behavior of mortar when exposed to natural weathering.

Another unexpected development of the natural weathering data should be noted. It will be seen from Table IV, that 12 out of 15 of the mortars showed higher expansions in natural weathering for the lower water-cement ratios. The three exceptions, Nos. 4, 13, and 17, were all high in clay and silt, though No. 18 had higher clay and silt content than No. 4. Accepting expansion as evidence of disintegration, the behavior of these specimens indicates that the water-cement ratio is not as reliable an index of durability as has formerly been supposed. A similar result has been obtained on a group of specimens, varying chiefly in the brand of cement, which were exposed to natural weathering. For this group, beams made with a water-cement ratio of 6.1 gal. per sack showed greater expansion than specimens made with a water-cement ratio of 7.89 gal. per sack in 7 cases out of 9.

So far as the loss in weight is concerned, all mortars but one showed higher losses for the high water-cement ratio mortars. The exception was the haydite mortar No. 16. Failure in this mortar is unlike that of any other. The specimens split but do not crumble or scale. This action is to be expected because of the very high absorption and therefore great internal expansion of the aggregates when freezing occurs. It will be seen from sections (*h*) and (*k*) of Fig. 4 that the high water-cement ratio specimens of aggregate No. 16 were the second lowest in resistance to weathering when expansion was used as a criterion, but were quite resistant (sections (*i*) and (*l*)) when the loss in weight was used as the criterion.

In Table IV data are given for a few beams which were placed outside on metal racks so that drainage would occur freely. For this natural weathering exposure it will be seen that all specimens except one, No. 15, show shrinkages. Except for some sand-streaking, these beams apparently have not been injured by the exposure. Comparison of the data for companion specimens stored in 1 in. of water will show the important influence of the exposure condition on the data from natural weathering. Strength is popularly regarded as a criterion of durability, the durability increasing with increase in strength. However, the data plotted in Fig. 4 show this widely-used rule to be unreliable when differences in strength are the result of the use of different fine aggregates and water-cement ratios differing by not more than 1 to 2 gal. per sack. Referring to sections (*a*) and (*d*) of Fig. 4, no relation is shown between the compressive strength and the resistance to cycle *H* when the expansion is used as a criterion. When the loss in weight is used as a criterion (sections (*b*) and (*e*)) a fairly definite relation exists between the compressive strength and cycle *H*, except for specimens Nos. 13 and 14. When cycle *P* is used (sections (*c*) and (*f*)) there is again no relation which is

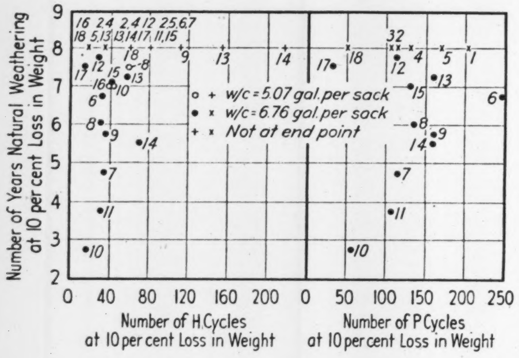

FIG. 5.—A Comparison of the Effects of Natural Weathering and the Laboratory Cycles on 2-in. Cubes.

true also for resistance to a 10 per cent sodium sulfate solution (sections (*g*) and (*j*)).

Referring to sections (*h*) and (*k*) of Fig. 4, it will be seen that strength gives no indication of the resistance to natural weathering if the expansion of beams is used as the criterion. A somewhat closer examination is required for sections (*i*) and (*l*). When the loss in weight of cubes is used as the criterion, the data of sections (*i*) and (*l*) show that on the average specimens of higher water-cement ratio and therefore lower strength may be expected to have less resistance to natural weathering.

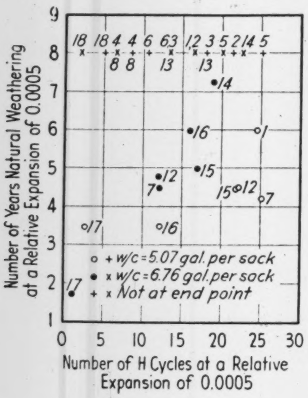

FIG. 6.—A Comparison of the Effects of Natural Weathering and One Laboratory Cycle on 2 by 2 by 10-in. Beams.

Though it is possible that trends may develop in sections (*i*) and (*l*) when more data are obtained, the wide spread in durability for approximately equal strength indicates little hope of a useful relation.

As a result of a previous investigation[9] of mortar in which the brand of cement and the water-cement ratio were the variables and the fine aggregate was a constant, it was concluded that "For a given cement or for the average of a number of cements, the resistance of mortars to artificial freezing and thawing increases with increase in compressive strength, but for mortars of the same water-cement ratios and consistency made from different cements, this relation does not hold and for high water-cement ratios may be reversed." Hence, taking the data of both investigations into account, it is evident that strength should not be used as an acceptance test for durability, since it would necessarily be applied to specimens differing in aggregate, brand of cement, water-cement ratios, and other factors.

Indirect comparisons have been made

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[9] C. A. Hughes, "The Durability of Cement Mortars: The Cement and Method of Testing Major Variables," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 511 (1933).

previously of the relative effects of laboratory tests and natural weathering. Figures 5 and 6 give a direct comparison using equal end points. It is likely that some modifications in these figures will result when data from the natural weathering is obtained which will permit the use of 30 per cent loss in weight or relative expansions of 0.003 as criteria. Referring to the left-hand section of Fig. 5, no relation exists between natural weathering and cycle *H* and, so far as the authors are concerned at least, no further time need be lost pursuing this particular will-o-the-wisp.

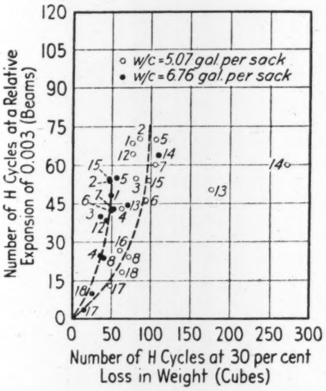

FIG. 7.—Relations Between Resistance of Mortars to Cycle *H* Measured by Use of 2 by 2 by 10-in. Beams and by Use of 2-in. Cubes.

In the right-hand panel of Fig. 5, a very nice relation is shown between natural weathering and cycle *P* using weight loss as a criterion for the specimens which reached an end point in 6 yr. or less. However, when the data for all specimens are considered from the viewpoint of setting some number of *P* cycles as a specification limit, it is at once evident that cycle *P* is of no more value than cycle *H* as a criterion of resistance to natural weathering. Essentially similar remarks apply to the data of Fig. 6 in which resistances of beams to natural weathering and to cycle *H* are compared.

It has been evident throughout the discussion of the data that the durability rating given any mortar would depend not only on the cycle used but also on the size and shape of specimen and on the method of determining the end point. In Fig. 7 fairly definite trends are shown between the resistances of specimens to cycle *H* when measured by an expansion end point on beams and a weight end point on cubes. However, if the specimens of higher resistance only are considered, it is found that considerable variation in expansion data can occur for a limited range in weight data. Figure 8 shows that there is no relation between the data from the expansion of beams and the data from the loss of weight of cubes that were exposed to natural weathering. Consequently, it appears that the size and shape of the specimen and the method of obtaining the end point are important considerations in devising durability tests for control purposes.

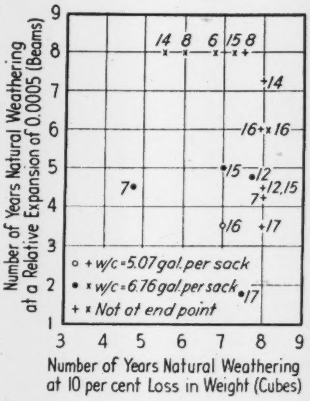

FIG. 8.—The Resistance of Mortars to Natural Weathering as Measured by Use of 2 by 2 by 10-in. Beams and by the Use of 2-in. Cubes.

SUMMARY

Because the number of variables was large relative to the number of specimens and because the natural weathering data are incomplete, it is thought that conclusions more definite than the statements of the synopsis are not justified.

Lack of useful correlation between the data of the two laboratory freezing-and-thawing tests of mortars used in this investigation and the data from the natural weathering of mortars is but additional evidence of the desirability of fundamental research in durability.

The need for laboratory control tests for durability is evident in numerous fields of the concrete industry. In the search for such control tests, the authors believe that success, as measured by correlation with service exposure, will be found in the slower cycles.

Acknowledgments:

Acknowledgment is gratefully made to the Graduate School for funds used in the laboratory tests and to the Engineering Experiment Station for funds and assistance in maintaining observations on the natural weathering specimens.

A DEVICE FOR STUDYING THE WORKABILITY OF CONCRETE

By T. C. Powers[1] and E. M. Wiler[1]

Synopsis

The terms *workability* and *consistency* refer to complex qualities not capable of direct measurement. They represent the combined effects of several properties, the most important of which are believed to be the following: (1) the yield value; (2) the characteristic force-flow relationship; and (3) the capacity for plastic distortion, that is, the maximum possible distortion without dilatation. A machine has been developed that is capable of distorting a plastic mass at any desired rate and of giving an automatically recorded graph of the acting force and of the rate and degree of distortion. This device is designed for a laboratory study of paste, mortar, and concrete mixes.

The purpose of this paper is to describe a new apparatus for laboratory studies of the workability of concrete and similar granular, plastic materials. Before describing the device itself it is necessary to review certain features of the physics of flow and plastic deformation, much as was done when the basic principles of its design were first conceived.

Definitions of Terms:

So much has been written about the workability of concrete with so many different definitions and connotations for the same words that it seems necessary to establish at the outset the ideas which the authors hope certain words will convey. Such words are: *workability*, *consistency*, *plasticity*, *cohesiveness*, *segregation*, *viscosity*, *mobility*, and *yield value*. In addition, certain new or less used terms must be considered: namely, *dilatancy* and *the capacity for plastic distortion*. A common understanding concerning the meaning of these words cannot easily be reached through a list of definitions. It seems better to discuss the facts and theories to which they pertain so that their meaning will be made clear by the context.

The term "workability" is qualitative only; it represents a quality that cannot be measured in the fundamental units of mass, length, and time. Therefore, it has no universally recognized, definite meaning; it is intrinsically vague. This is illustrated by the following definition, proposed in another paper by one of the present authors.[2]

"Workability is the combined effect of those properties of fresh concrete that determine the amount of internal work required for placement and compaction, and that determine the resistance to segregation." To search for a means of directly measuring workability, so defined, is clearly futile. There is no conceivable fundamental unit of measure for such a complex quality.

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[1] Assistant to the Director of Research, and Assistant Physicist, respectively, Portland Cement Assn., Chicago, Ill.

[2] T. C. Powers, "Workability of Concrete," to appear in the revised Report on Significance of Tests of Concrete and Concrete Aggregates to be published by the American Society for Testing Materials.

Likewise, as Breyer and Green[3] have said, the word consistency takes on many meanings according to context. It can be and has been applied to everything from gases to crystalline solids.

Despite the complex nature of these words, they undoubtedly reflect a sensory perception of certain combined qualities which separately should be measurable in fundamental units. Because of this it is proposed to deal with those properties that seem to be components of the qualities recognized as *workability* and *consistency* without attempting to decide the exact degree to which each fundamental property is concerned with each of these two qualities.

The term plasticity can be more definitely defined than the terms just discussed. Plasticity will be used here only with reference to comparatively thick suspensions of granular solids in fluid, a general classification in which cement-pastes, mortars, and concretes can be placed. Such suspensions take on properties that are characteristic of fluids at one extreme and of solids at the other. How this is true can be made clear by first considering the nature of fluid-flow.

Flow of Fluids:

When a fluid such as water is caused to flow slowly through a tube, a frictional resistance is experienced. Likewise, if it flows as a sheet over a solid surface, similar resistance is found. This resistance may be regarded as arising primarily from the differences in the velocities of various parts of the stream of liquid; the velocity is zero at the solid-liquid boundary and increases with the distance from the surface. In a tube the flow may be visualized as the movement of coaxial cylinders of infinitesimal wall thickness, the center cylinder having maximum velocity while the outer one remains fixed with respect to the solid boundaries.[4] With liquid flowing over a plane surface the flow may be regarded as the sliding of sheets or laminations over each other, the sheet at the solid-liquid boundary having zero velocity and that near the air-liquid boundary, maximum velocity. In both cases, continuous flow is a process of continuous shearing of the liquid, and the observed resistance to flow is the effect of resistance to this laminar shear.

A characteristic of the liquid state is that shear can be produced by any force however small and the rate of shear for so-called simple, or Newtonian, liquids (including water) is directly proportional to the applied force. The proportionality factor between force and rate of shear differs with the nature of the liquid and with temperature. It is called the *coefficient of viscosity.*

Flow of Suspensions:

When a fluid is mixed with finely divided solids, forming a suspension, the viscosity of the system as a whole changes little from that of the liquid alone, if the proportion of solids is small. At higher concentrations the viscosity of the mixture becomes noticeably greater than that of the liquid alone, and the resistance to shear (that is, to flow) increases very rapidly with concentration when the concentrations are such as to produce mixtures commonly recognized as pastes. When the concentration of granular solids is made high enough, perhaps with the aid of pressure, the resistance to shear approaches that of the solid particles themselves. As said before, in the intermediate condition between fluid and solid, the mixture behaves partly as a fluid and partly as a solid.

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[3] F. G. Breyer and Henry Green, "Introduction: A Discussion on Consistency," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 432 (1923).

[4] For the purpose of this discussion it is not necessary to consider turbulent flow; viscous flow is assumed throughout.

For this discussion attention can be centered on the comparatively thick suspensions represented by pastes, mortars, and concretes. It is convenient to consider pastes alone at first.

If the solids in suspension are independent of each other—well dispersed—the paste is likely to behave much like a fluid, although the relation of rate-of-flow to force may not be linear.[5] If, on the other hand, the particles are highly agglomerated as are the calcium hydroxide particles in a lime putty or the cement particles in a cement-water paste, the paste then has some of the properties of a solid. This is sometimes denoted by the ability of the mixture to hold its shape against small forces. For example, on withdrawal of a trowel the paste may show standing spines or ridges on its surface. Such materials under test show that some minimum force must be exceeded before flow starts. This minimum force is called the *yield value.* It is believed that the response to forces smaller than the yield value, if the force is applied for only a very short interval of time, is that of an elastic solid.

Such solidity may be apparent immediately after mixing or it may develop with time. When the latter is the case, the solidity is said to be the result of what is called thixotropic set. When the material is of this nature, the yield value is not a fixed property of the mixture but depends to an important degree on the history of the sample. The previous history of the sample (that is, the method of mixing and the time-interval since mixing, etc.) probably also influences the yield value of nonthixotropic pastes, but few or no confirming data are at hand.

As said before, mixtures having yield values probably behave much as true solids under very small stresses. However, unlike true solids, their response to stress is regulated in an important way by the rate at which the stress is applied and by the interval of time over which a given stress is maintained. With concretes and mortars in the solid state, this has been shown to be true; it has not yet been demonstrated for the plastic state.

A mixture which, by virtue of its yield value, is capable of withstanding some small stress without giving way and which also will distort without rupture or change in volume under a sufficiently large force, exhibits behavior which clearly meets the requirements of a common dictionary definition of the adjective "plastic": "capable of being deformed continuously and permanently in any direction without rupture."

By some definitions the material must also be capable of holding its new shape after being distorted if it is to be classified as plastic; but this is a very indefinite specification, for it depends too much on the particular shape and position produced. For example, a plastic mixture might easily retain its shape if rolled into a thin sheet on a horizontal surface, but it might fail if it were made cylindrical. A material showing any finite yield value would be able to hold some shape in some position. Therefore, it seems clear that a material showing a yield value, however small, meets part of the definition of a plastic.

When a plastic material is stressed beyond its yield value, distortion takes place, the amount and rate being characteristic of the material. Such distortion may be considered to be flow in that it involves the same type of shear as fluid-flow and the energy used is apparently not stored as potential energy recoverable on release of the force; that is, distortion is apparently inelastic. With some materials, under some conditions of test, the differential of flow-rate with respect to force is constant and thus is a factor analogous to the coefficient of viscosity, that is, when rate of flow is plotted against force, the result is a straight line. But, since the line does not pass through the origin, the force-flow ratio for such data cannot properly be called the coefficient of viscosity; unlike viscosity, it varies with the magnitude of the acting force. Bingham[6] has called the slope of such a curve the *coefficient of mobility*.

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[5] For a description of the many possible types of flow see "Elasticity, Plasticity and Structure of Matter," by R. Houwink, p. 10, Cambridge University Press (1937).

Bingham regarded the yield value (or "friction," as he called it) together with mobility as constituting a complete measure of plasticity. However, he did not measure the yield value directly but assumed it to be identical with a value found by extrapolating the straight line representing the relationship of force to flow obtained under test conditions producing relatively high rates of shear. It is now believed that the value thus obtained is seldom, if ever, the true yield value.

As a matter of fact, there are few, if any, direct experimental data which bear out the usual assumption that relatively thick, plastic suspensions give test results like those on which Bingham's nomenclature is based. Certainly, the experiments of Norton[7] with mixtures of clay stiff enough to be molded into cylinders show no evidence of such behavior. On the contrary, the resistance of such materials to distortion resembles very closely that of a true solid when stressed up to and beyond its yield point; at small strains the required stress is proportional to strain and at higher degrees of distortion stress further increases with strain but is not proportional to it.

Roller,[8] using a different technique, also has obtained similar results for a wide variety of relatively stiff, plastic materials. That is, he too found that stress must increase with the degree of distortion. It should be noted carefully that this is distinctly not the behavior of a fluid, nor of a plastic as defined by Bingham, for with fluids and with plastics so defined, the required force is entirely independent of the degree of distortion and depends only on the rate of distortion. Roller's findings show that the force required for distortion of pastes, greases, etc., stiff enough partially to hold a cylindrical shape, depends almost solely on the degree, and hardly at all on the rate, of distortion. Data to be reported later in this paper give some support to this conclusion.

It seems that many of the conclusions that have been reached heretofore concerning the behavior of plastic materials do not apply to pastes or other thick, granular suspensions. Nevertheless, it is evident that the quality known as plasticity must depend on the yield value of the material and the manner in which the material responds to forces exceeding the yield value. However, it does not follow that these two factors are sufficient to describe completely the degree of plasticity possessed by a given material. To accept such a conclusion would be to overlook one of the most important factors that cause certain materials to be considered as a separate class, "capable of being deformed continuously and permanently in any direction *without rupture*." This qualification "without rupture" is to be noted especially. It implies not only that any given solid is plastic while it is deforming continuously, inelastically, and without rupture under stresses greater than its yield value, *but also it implies that when the same material ceases to deform in this manner it likewise ceases to behave as a plastic.* Thus, not only are the yield value and the characteristic response to forces greater than the yield value involved in the concept of plasticity but also a limit on the inherent *capacity for plastic deformation* is an essential part of this concept. That is to say, the plasticity of a body cannot be described in less than three terms, as follows:

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[6] E. C. Bingham, "Fluidity and Plasticity," McGraw-Hill Book Co., Inc., New York, N. Y. (1922).

[7] F. H. Norton, "An Instrument for Measuring the Workability of Clays," *Journal*, Am. Ceramic Soc., Vol. 21, pp. 33-36 (1938).

[8] P. S. Roller, "Plastic Flow of Dispersions and a New Approach to the Study of Plasticity," *Journal of Physical Chemistry*, Vol. 43, pp. 457-489 (1939).

1. The force required to start deformation—the yield value.
2. The characteristic response to forces exceeding the yield value—the stress-flow relation.
3. The capacity for plastic distortion.

Dilatancy:

The limit of plastic distortion is marked by the beginning of structural failure, a rupture of some sort, or by the phenomenon known as dilatation. Dilatation may be manifested in several forms: it may be observed easily by forming a ball of neat cement of about normal consistency and then twisting the ball so that one hemisphere tends to rotate with respect to the other. The twisting is accomplished with little effort up to a certain maximum angle at which the required effort for further distortion increases sharply. Any distortion beyond the point where the required force increases is accompanied first by a drying of the surface in the zones where the strain is greatest and then by rupture. The drying at the surface is caused by an increase in the volume of the paste without a corresponding increase in water content, that is, it is caused by dilatancy defined by the dictionary as "having the property of increasing in volume when changed in shape owing to increase of the space between the particles."

The necessity for a wider spacing of the particles arises in this case from the fact that when compacted to the degree represented by normal consistency each cement grain, though separated from its neighbors by water, nevertheless has but a limited range of free movement without making contact with a neighboring grain. When distortion is produced, shear within the mass causes adjacent layers to slide one over the other. By regarding such layers as having infinitesimal thickness, the grains, having finite dimensions, tend to lock together adjacent layers, or shear-planes, and the locking becomes positive as soon as the grains have made contact in attempting to slide past each other. If distortion is forced beyond this limit, the particle interference described above causes the mass to expand: it causes dilatation.

Another frequently cited example of dilatancy is that which was given by the originator of the term, Reynolds, in 1885.[9] One of Reynolds' observations is that of the behavior of wave-beaten beach sand which whitens and appears to become dry with each footfall but takes on its original appearance when the pressure is released. This is caused by the displacement of closely packed grains in such arrangement that only slight deformation can take place without dilatation.

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[9] Osborne Reynolds, "The Dilatancy of Media Composed of Rigid Particles in Contact," *Philosophical Magazine*, Vol. 20, p. 469 (1885); "Dilatancy," *Nature*, Vol. 33, p. 429 (1886).

Freundlich and Röder[10] describe another example, given originally by Wo. Ostwald, in which certain fine powders, starch for example, when mixed with a limited amount of water and stirred slowly behave like a viscous liquid. When the speed of stirring is increased, the mass becomes seemingly dry but recovers its fluidity again when the rate is decreased. This experiment illustrates the important fact that under some circumstances dilatancy involves the element of time as well as the properties of the components of the mass. That is, a mass which may be capable of a given amount of plastic deformation at a low rate of deformation may at a sufficiently high rate exhibit marked dilatancy, apparently because the particles do not have time to find the paths in which free movement might have taken place.

In general it may be said that in any mass in which the particles are so spaced that they have restricted freedom for movement, the mass will become dilatant at some *degree* of distortion. Furthermore, in a suspension of independent particles, even if it is possible geometrically for any particle to find a free path through the mass, the system may become dilatant if the *rate* of distortion becomes sufficiently high.

Experience with pastes indicates that a granular plastic can exhibit a tendency toward dilatation without actually becoming dilatant. At some degree of distortion a definite interlocking takes place, but on increasing the acting force, the mass continues to deform without expansion. Such behavior could be explained by assuming that the interlocking is not between individual particles but between flocs which have relatively little structural strength. The continued plastic distortion following the initial interlocking is made possible by the distortion or rupture of these flocs. It is possible also that dilatation may not follow the initial interlocking of particles because continued increase in force may bring about a change to a less dilatant configuration of the particles.

Summary:

From the foregoing discussion it should be evident that in this paper the terms workability and consistency are to be regarded as qualitative attempts to describe in a word the combined effects of several definable physical properties of granular, plastic materials. In a study of the fundamentals of plastic mixtures it is hopeless to attempt to measure such dimensionless qualities directly. However, the qualities in question involve properties such as viscosity, yield value and the force-flow relationship that are amenable to quantitative measurement. These factors together with the capacity for plastic deformation—that is, the degree of distortion at which the tendency toward dilatation develops—combine to give a quantitative measure of plasticity. The term "plasticity" embodies most, but not all, of the concepts of workability and consistency.

Resistance to Segregation:

The discussion up to this point has been concerned only with those properties of concrete that influence the "internal" work done in placing concrete. A complete study of workability requires also consideration of the latter part of the definition of workability given previously, that is, the qualification concerning the ability to resist segregation. The term "segregation" has been used in various places to mean separation of water, separation of aggregate sizes, or departures from homogeneity in any respect whatever. Segregation of water is the phenomenon elsewhere called bleeding. The same factors that control bleeding undoubtedly influence workability to a marked degree, but the authors are doubtful that bleeding should be considered as an independent variable in a study of workability.

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[10] H. Freundlich and H. L. Röder, "Dilatancy and Its Relation to Thixotropy," *Transactions*, Faraday Soc., Vol. 34, pp. 308-316 (1938).

As to the segregation of aggregate sizes, it can be seen readily that a mixture distorted to the point of dilatation is very likely to segregate on further distortion. In fact, the distortions produced in most mixers must be such as to maintain some degree of segregation throughout the period of mixing. Therefore, it is believed that the capacity for plastic distortion has an important bearing on the ability of a mixture to resist segregation.

Finally, it might be necessary to consider the cohesiveness of the paste part of the mix as an independent variable controlling resistance to segregation. However, here also there is reason to believe that it may not actually be an independent variable because cohesiveness seems to depend on the specific surface of the particles, the shape of the particles, and the physicochemical relationship between the solid and the liquid phases. These are the same factors that control flocculation, yield value, and probably the force-flow relationships of the paste. Consequently, there is hope that, when the factors determining the degree of plasticity are measured satisfactorily, all of the factors that characterize workability will have been adequately taken into account.

It is to be emphasized that the theories and opinions expressed in the foregoing discussion are on the whole not to be considered as facts accepted by the authors but as a working hypothesis that is being used in the study now under way. No doubt occasion for correction and change of emphasis will arise as the experimental work proceeds.

DESCRIPTION OF THE NEW APPARATUS

Based upon considerations such as the foregoing, an attempt was made to produce a device that would measure the three factors believed to be most important to workability. Several machines were built and found to be unsatisfactory for one reason or another. Finally, a machine was developed which utilizes the principle found in the Couette or the McMichael viscometer. Although the machine is not satisfactory in all respects, it meets the major requirements of the experimental work for which it was developed—a study of pastes and concrete mixes.

Figure 1 shows the general appearance of the machine. Figure 2 is a close-up view of the essential parts. Cylinder *A* is 12 in. in diameter and 8 in. deep. *B* is a hollow drum 8 in. in diameter and 3 in. deep, with both top and bottom closed. The rectangular object is an automatic recorder. Both the drum and the recorder are fastened rigidly to the shaft. The test-sample (paste, mortar or small-aggregate concrete) fills all the space under and around the drum.

The drum in its normal position is held about 4 in. above the bottom of the container. By this arrangement the test is made only on the top part of the sample and the results are therefore not immediately affected by the layer of sediment (the "compression zone"[11]) which begins to develop as soon as the sample is in place.

The sample may be tested in several different ways. When the machine is assembled as shown in Fig. 2 and the sample is in place, the operation is as follows:

By means of the motor shown in Fig. 1, container *A* is caused to turn around its

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[11] See *Bulletin 2*, Portland Cement Assn., pp. 3-12, 84-85 (1939), for a discussion of the development of a "compression zone" during the period of sedimentation (bleeding).

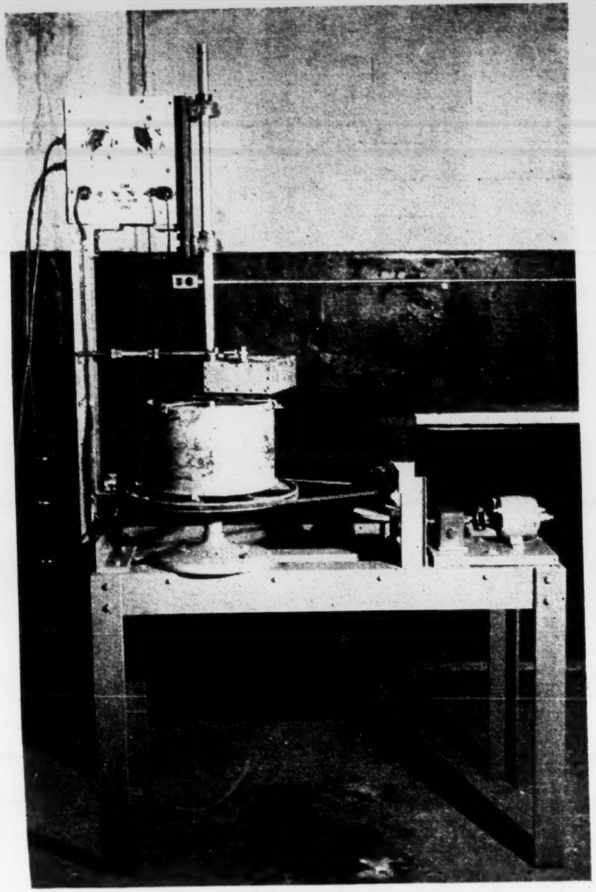

FIG. 1.—General View of Device for Studying Workability.

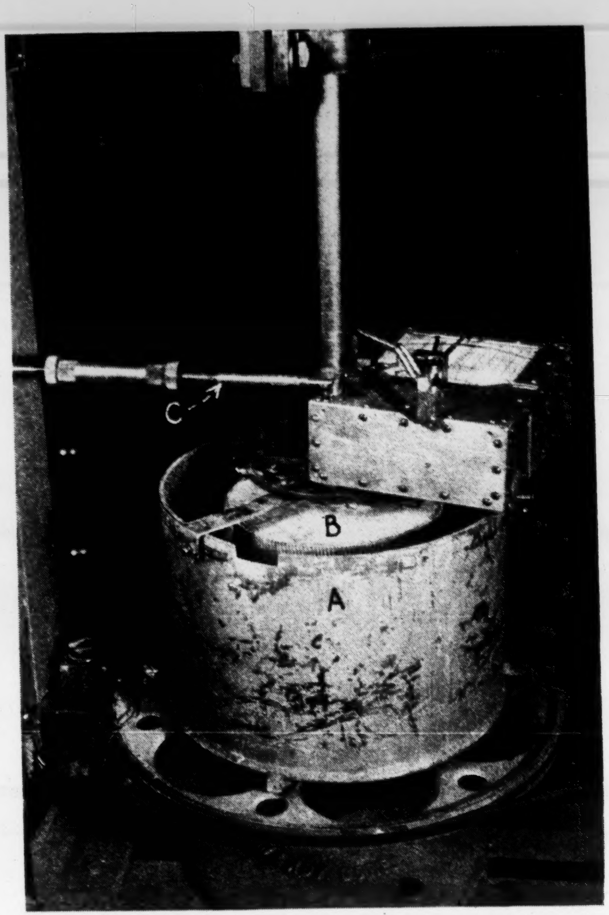

FIG. 2.—Close-Up of Sample Container and Automatic Recorder.

A = Sample container.
B = Drum.
C = Stay-bar.

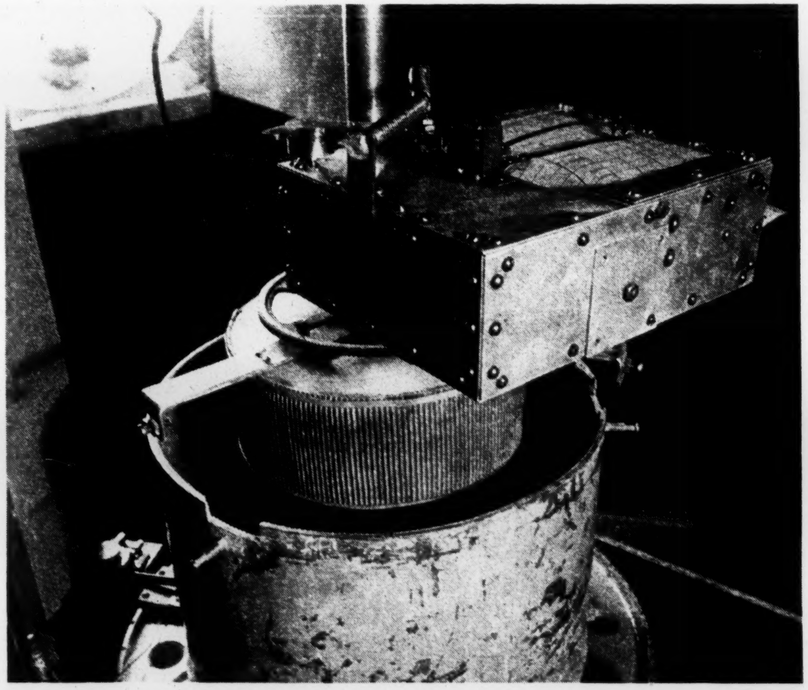

Fig. 3.—Inside Drum Lifted Above Its Normal Position to Show Its Dimensions. It may be lifted to a still higher position to facilitate placing or removing the test sample.

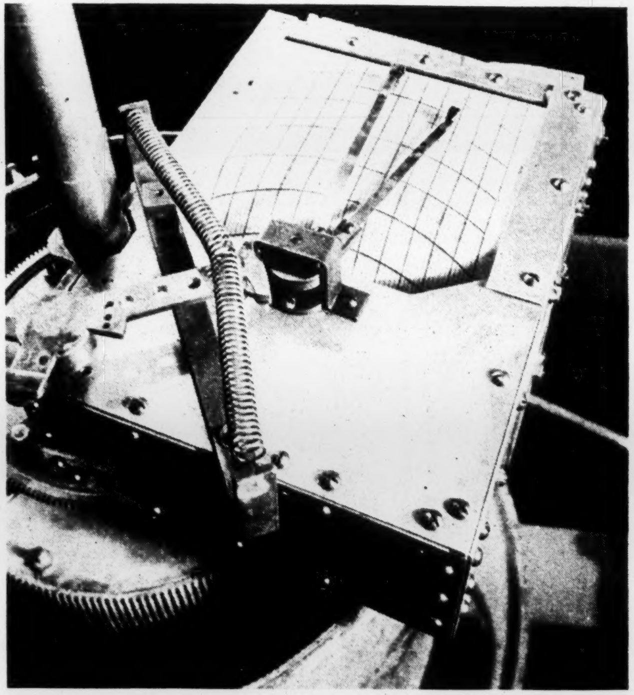

Fig. 4.—Close-Up View of Recorder.

vertical axis. The rotation of A (Fig. 2) reacts on drum B through the material filling the space around the drum, producing a torque tending to rotate the drum in the same direction. The rotation of the drum is resisted by the stay-bar C. (See Figs. 1 and 2.) One end of the stay-bar is attached by a hinged joint to the vertical member of the frame and the other is connected through another hinged joint to the lever-and-spring arrangement shown best in Fig. 4. Because of this arrangement, the rotation of the drum is opposed by the spring. Any displacement of the spring is registered by one of the two pens shown in Fig. 4 on graph paper moving one inch per second.

Since the drum is not permitted to follow the rotation of the container, the material in the annulus is distorted. A measure of the distortion is registered on the moving paper by the second pen, which is actuated by a suitable arrangement of gears that are caused to rotate by any angular change of A with respect to B. Slip between the sample and the apparatus is prevented by the ribbed-rubber coverings shown.

The actual distortion of the material in the annulus is not uniform but varies from a minimum at the outside to a maximum at the inside. The recorder indicates only the change in position of the boundaries and hence it registers the integral of the unit distortions across the annulus. The relationship of the recorded value to the actual distortion at any point in the annulus is therefore determined by the dimensions of the machine. However, in the discussion to follow, the angular displacement of the boundaries as indicated by the recorder will be considered to represent the actual distortion of the material.

With the ordinary rotating-cylinder type of viscometer, the rotation is continuous throughout the test. When this procedure is followed using pastes or mortars, normal distortion of the sample is not produced. Instead, after a fraction of a revolution, the sample fails in shear near the surface of the drum, the region of highest stress. After failure occurs, the bulk of the material in the annulus develops little distortion, if any, when the rotation is allowed to continue. Instead, the additional distortion occurs entirely within a very thin zone ($\frac{1}{8}$ in. or less) adjacent to the curved surface of the drum. When this condition of extreme non-uniformity of distortion is reached, it is manifestly impossible to interpret measurements of total distortion or rates of distortion in terms of the characteristics of the sample as a whole. That is, it appears neither feasible nor correct to attempt to measure the plastic properties of a mix by observations on its behavior during continuous rotation of the container. The results obtained from continuous rotation may not be without significance however, for the maximum torque developed indicates the strength of the sample in shear, and the torque necessary to maintain rotation is determined by the frictional resistance developed in the thin zone of failure.

It has been suggested that the strength in shear is a significant characteristic of a mix. If—on further study—this should prove to be true, the machine can be used to obtain this information.

Because it is unnecessary and undesirable to allow continuous rotation of the container when measuring the plastic characteristics of a mixture, the machine was so constructed that distortion can be stopped at any point or the container can be caused to oscillate automatically through any desired arc. Back of the control board (see Fig. 1) is a switching mechanism that can, if desired, be used to produce automatically a series of twelve oscillations, the rate of distortion changing every second cycle. By this means the effect of varying the rate of distortion can be recorded quickly.

TYPICAL TEST RESULTS

In Fig. 5 the results of tests made on a viscous liquid, glucose, are given. In obtaining these data the container was caused to rotate continuously with the distortion-registering mechanism disconnected. The diagram shows that following a period of acceleration the torque remains constant. The value of the registered torque depends on the rate of rotation and on the viscosity of the glucose. When these values for torque are plotted against the rate of rotation, a straight line passing through the origin is obtained, as shown in Fig. 6. This result shows that the instrument correctly registers the characteristics of a fluid.

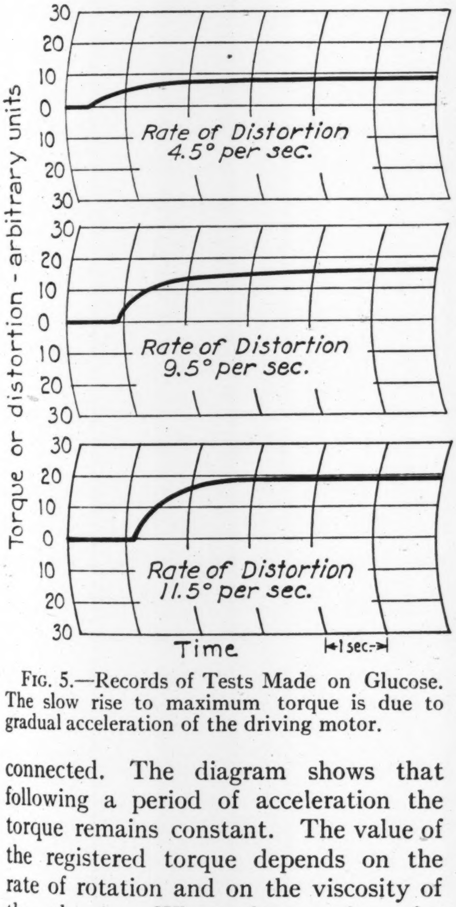

FIG. 5.—Records of Tests Made on Glucose. The slow rise to maximum torque is due to gradual acceleration of the driving motor.

Figure 7 is a typical record of a test on portland cement paste with the machine set to oscillate as described above. At time A the pens registered zero torque and distortion. Then in the interval AB (about $2\frac{1}{2}$ sec.) the distortion proceeded at a nearly constant rate and the torque acting on drum B (Fig. 2) increased at a steady rate. Note that all changes away from the zero line, whether above or below, are to be considered as positive. The two pens move oppositely.

Torque and distortion reached their peaks simultaneously; the seeming lag of the torque curve is due to the fact that the pens are in tandem.

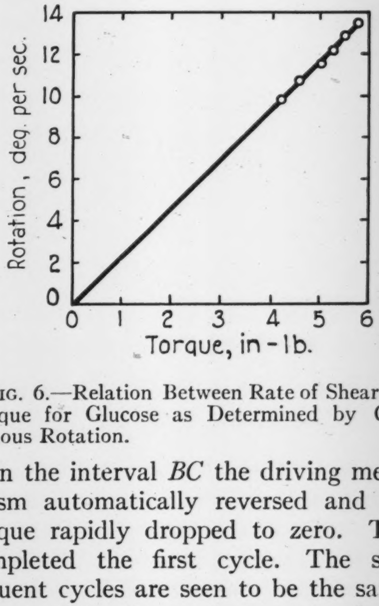

FIG. 6.—Relation Between Rate of Shear and Torque for Glucose as Determined by Continuous Rotation.

In the interval BC the driving mechanism automatically reversed and the torque rapidly dropped to zero. This completed the first cycle. The subsequent cycles are seen to be the same, except that the total amount of distortion is twice that of the initial distortion. This is a consequence of starting the test at the center of the range of oscillation.

Torque-Distortion Relationship:

To facilitate interpretation, data in the form shown in Fig. 7 are reduced to that shown in Fig. 8. This diagram shows the relationship between torque and distortion for samples that were distorted at a constant rate. Two kinds of samples are represented: a 1:2 mortar gaged to a 2-in. slump and the same kind of mortar gaged to a 3-in. slump (6-in. cone). For each mortar, two curves are given, *A* and *B*, *A* representing the relationship for the first distortion after the sample was placed in the apparatus and *B*, the fifth distortion. The upper of the two curves *A* will be considered first.

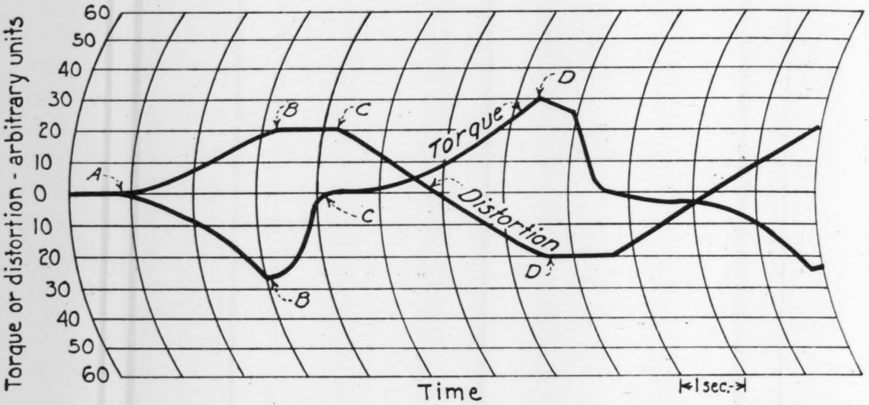

Fig. 7.—Typical Record Obtained When the Machine Is Set to Give Oscillations of About 10 deg. on Either Side of the Original Position. Curves for pastes, mortars and concretes all have the same shape. This graph represents a neat paste—water-cement ratio = 0.27 by weight.

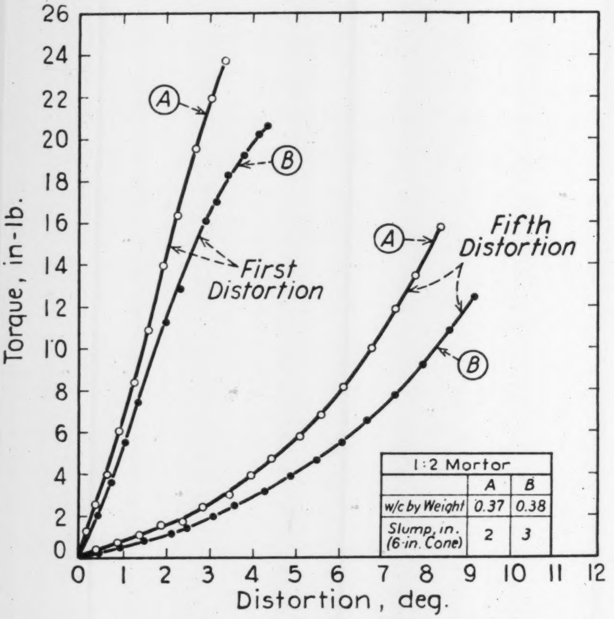

Fig. 8.—Typical Relationship Between Torque and Distortion.
Rate of Distortion: First deformation, 3 deg. per sec.; fifth deformation, 5½ deg. per sec.

This curve shows that when the mix is distorted the torque increases almost proportionately with the distortion. This is a remarkable result in that it indicates that a plastic mortar has stress-strain characteristics superficially like those of a solid. A curve such as *A* cannot be constructed for a fluid, because in fluids the force required to produce flow is independent of the amount of distortion produced within the fluid by that flow and depends only on the rate of distortion. With solids the opposite is true: The force required is practically independent of the rate of distortion and depends almost wholly on the amount of distortion. Thus, curve *A* of Fig. 8, showing that in mortar mixes the required force does depend upon the degree of distortion, together with other data (not represented in the graph) showing clearly that the rate of distortion has very little influence on the relationship shown, indicates that the behavior of a mortar is predominantly like that of a plastic solid and not at all like that of a fluid. Cement pastes and lime putties show the same kind of characteristics.

The peculiar behavior of this type of material is further exemplified by the large change brought about by repeated distortions. This may be seen in Fig. 8 by comparing the relative positions of the curves for the first and fifth distortion. During the operation of the machine it can be observed that the greatest resistance is offered during the first distortion. The second distortion can be produced with much less resistance and subsequent distortions result in further, though smaller, reductions in resistance. A tentative explanation of this effect can be offered.

It seems that during the first distortion the particles, in their initial, random arrangement, offer the maximum of particle-interference. On subsequent distortion the particles have become somewhat rearranged and oriented so that they have more freedom for movement. The practical consequence is that the resistance to distortion of a given mix depends not only on the composition of the mix but also on the degree to which it has previously been distorted in the same or opposite direction. (Incidentally, this fact is utilized by experienced workmen who habitually take advantage of the effectiveness of "puddling" or "quaking" the mass to promote flow.)

The fact that mortar and, undoubtedly, concrete do not behave at all like a fluid at any stage of distortion brings out clearly the futility of attempting to apply many of the earlier ideas in the interpretation of data such as those presented above. It therefore becomes necessary to establish some of the fundamentals of this type of plastic flow before the new apparatus can be intelligently applied to the study of gradation and mix design for which it was intended. Experiments along this line are now under way.

DISCUSSION

MR. C. A. G. WEYMOUTH[1] (*by letter*).—An important advance in our knowledge of workability of concrete mixtures can be expected from tests made with the new machine described in this paper. It is the writer's opinion that it more truly reproduces essential movements that occur in placing concrete than any of its predecessors, even than the remolding test machine.[2] The alternations of distortions reproduce the effect of waves of motion and accompanying surges of force that simultaneously affect all parts of the plastic mass. Vertical jigs, used in the remolding test, do not occur in practical concrete operations, and, besides, jigging causes heavy pieces to become unduly concentrated within the ring clearance at the bottom of the apparatus and this clouds the interpretation of the test data. In the present device the only applied forces are horizontal.

While this machine may handle a wide range of consistencies, the writer believes that some important influences on plasticity which affect very stiff mixtures are absent in high-slump concretes. For this reason comparison of workability of mixtures should be made at equivalent relative consistencies. A distinctive difference in plastic states is recognized by both ceramists and soil engineers which should be applied to studies of plasticity in concrete. In a stiff cement paste the finest solids are so closely spaced and the water films are so thin that polarized water, solvated layers, molecular cohesion combined with molecular adhesion, all play a part in the cohesion of the aggregation of solids, but such a paste is not a true suspension of solids in water since the solids predominate. On the other hand, in a paste made soft enough by adding mixing water until it slumps appreciably, these finest particles are separated by water films too thick for molecular attractions to be transmitted across them from solid to solid. Such a paste, if plastic, can be classed as a suspension since the liquid phase predominates. Rhodes[3] divides the plastic state of soils into two parts when highly plastic clays are tested: a cohesive, but nonsticky, plastic state and a sticky, adhesive, plastic state. Practical concrete mixtures that may be transported and placed principally by gravitation (down chutes, through tremie heads, etc.) are in the adhesive plastic state.

This distinction is important since it is generally agreed that mixtures like concrete have plasticity because the solids are in a state of suspension. Such solids are dispersed, separated, and held apart in opposition to gravitational pull or cohesive forces tending to agglomerate them. Most studies of plasticity have been made on cohesive relations in stiff pastes (cohesion implying strength—strength of molded specimens or strength in soils to support building loads). There is considerable confusion in the various explanations made to account for plastic suspensions because of the failure to recognize a critical difference in these plastic states. None that the writer has seen appears adequate. If the principles of plastic suspension were understood, it is likely that they would be found to influence the cohesive plastic state in addition to the cohesive forces usually thought most important. Such knowledge would aid in correct interpretation of test data furnished by this new machine.

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[1] Raymond G. Osborne Bureau of Tests and Inspection, Los Angeles, Calif.

[2] T. C. Powers, "Studies of Workability of Concrete," *Journal*, Am. Concrete Inst., Vol. 28, February, 1932, p. 425.

[3] E. O. Rhodes, "A Machine for Testing Highway Subgrade Soils," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 571 (1938).

A working hypothesis, developed by the writer to account for plastic suspension, is here offered to aid in correlating test data. It has provided him with a better understanding of concrete in field practice, and serves to explain the effects of thixotropy and dilatancy, terms discussed by the authors.

Plastic suspension of solids by water, a lighter medium, implies two things: (*1*) maintenance for a sufficient placing time of enough clearance between the solids to permit free movement along planes of shear and a sluggish interchange of portions of the mass while being molded, and (*2*) a vigorous retention of the water within the structure of the mix. Not only must clear water be kept from leaking from the cement paste, but cement paste must be retained within the voids of the sand in mortars, and mortar within the voids of the stones of concrete. Powers has shown[4] that bleeding of clear water begins immediately by a process of sedimentation when fresh concrete of high quality is placed and agitation has ceased. While this process is slow, the suspension of the solids is not positive or sufficient. It is evident that something besides specific surface and structure is needed. The writer finds that plastic suspension is the result of mild turbulence upon a proper structure of solids containing the requisite amount of colloidal material.

Coarse Structure of Concrete.—The structure of solids varying from fine cement to coarse stone is quite complicated when studied by the usual considerations of solidity ratio and void content. The real nature of such structure is disclosed, however, when it is examined as an aggregation of size groups of solids. Each size group, whatever the diameter of its particles, possesses an elementary structure in which the total voids are subdivided into a series of individual void spaces, called "void pockets." If the particles are large enough, of uniform size, and piled up in close packing, one can see that each pocket has a bulky volume outlined by the surfaces of surrounding stones with openings leading into adjacent pockets on all sides. A smaller particle just circumscribed in such a pocket would be much too large to pass through any of these openings. But a size group of particles is never "closed-packed" in a mixture with finer material as in concrete. The particles of the group are held apart from each other but they are still capable of forming the typical void pockets with bulky volumes and interconnecting openings, provided, of course, their average clearance is not too much for them to have a definite structure.

The void pockets defined by the particles of any size group are important because each contains its proportion of all the finer elements of the mixture of concrete. The freedom of movement of each such portion of matrix is held in check by the surfaces outlining the pocket and by the size of the orifices leading from it, and conversely the spacing of the particles of the size group is dependent upon this limited freedom of movement of its matrix.

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[4] T. C. Powers, "The Bleeding of Portland Cement Paste, Mortar and Concrete," *Bulletin 2*, Portland Cement Assn. (1939).

This structure of the size group persists even down to the microscopic cement solids.

Structure at the Finest Range of Solids.—The matrix filling the finest void pockets of definite character consists of water with its concentration of colloidal cement solids and hydration flocs.

When water is added to cement, the processes of hydration begin immediately. Anderegg and Hubbell found[5] the depth of hydration on medium cement grains to be 0.5 μ in 24 hr., but with cement flour practically all had hydrated in this time. With separate minerals, however, the depths of hydration on grains of tricalcium aluminate from 19 to 26 μ in diameter were 0.69 μ in 1 min. and 2.14 μ in 15 min. Similar results were obtained with tricalcium silicate. While evidence that clinker grains disintegrate in the presence of water into separate minerals was not found, it is likely that the rate of hydration of the finest and, therefore, the softest cement particles is very rapid. The concentration of hydrous material in the water films is undoubtedly increased by that rubbed off larger solids during the mixing period. The writer makes a distinction between the hydrated cement flocs that become coagulated, tenuous, and tuft-like and those grains of colloidal size not yet attacked by water.

Any structural air present would be in the form of microscopic bubbles and would be found within the smallest void pockets, tending to impede free movement of the flocculent elements into adjacent pockets. While clear water leaks, or "bleeds," through the orifices of these primary pockets when agitation has stopped in a concrete mass, the system of void pockets here outlined traps all fine elements during sedimentation and prevents "free settling." Powers virtually confirms this conception of void pocket structure in his statement[6] "in spite of the fact that the largest particles present may be a thousand times the size of the smallest . . . the particles of various sizes and densities do not settle independently, but all move in unison, the finer particles being carried down by the coarser ones."

Plastic Suspension.—As waves of motion pass back and forth through plastic concrete, surges of force are impressed upon each matrix in its series of void pockets, including those of the largest and finest sizes. These impulses are felt in all directions because the fluid phase is continuous. The result is a tendency for the elements in each pocket to move into an adjacent pocket in the direction of the surge. This tendency is resisted by the frictional contacts of the largest particles in the matrix at the orifices of the pockets. This resistance reacts on the particles of the size group forming the pockets and holds them apart, or "in suspension."

In the finest or primary void pockets, these surges roil the colloidal solids, some just not of colloidal size, together with the hydrous flocs and keep them turbid. Clots form across the openings of the pockets with each direction of motion and thus hold the water from leaking out similarly to blood at a cut that is matted with cobwebs. But when agitation of the mass stops, these matted clots loosen so that clear water can find normal bleeding capillaries.

An example of extreme turbulence and its power of suspension was provided by the failure of the St. Francis Dam in Los Angeles County. Great masses of mud, sand, gravel, and cobbles became mixed into a suspension by the sudden flow of escaping water. Huge blocks of concrete, one at least of which weighed more than a thousand tons, were carried more than half a mile downstream of small grade.

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[5] F. O. Anderegg and D. S. Hubbell, "Rate of Hydration of Cement Clinker," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 554 (1929).

[6] T. C. Powers, "The Bleeding of Portland Cement Paste, Mortar and Concrete," *Bulletin 2*, p. 14, Portland Cement Assn. (1939).

Effect of Gap Grading.—Ease of efflux of a matrix through the openings of its pockets depends upon the number of the largest particles of the matrix and the relation of their size to the size of these openings. When one size group is omitted in the grading just below the group forming the pockets being discussed, the largest size in the matrix is one half what it would be with continuous grading, and these particles are relatively less effective in blocking the orifices. Loss of suspension is the result.

Ordinary points of gap grading in concrete are those between the cement and sand, and between the sand and coarse aggregate, either of which causes a loss of plastic quality in the mix. Concretes which have these gaps only partially filled or without any material are watery in appearance and readily segregate. This is seen when the concrete flows down an inclined chute or by the settling of the mass in a concrete buggy as it is wheeled a short distance. Concretes with fully continuous grading of its solids and free from particle interference have a drier appearance, are more sluggish, but will flow down a chute without separation and the mix will stay "alive" and suspended even after being wheeled over long distances.

Particle interference of size groups, as this term is used by the writer, results in internal segregation of the conflicting groups during mixing. When a group of particles is too closely spaced in a mix, the next smaller particles cannot be uniformly mixed into the void pockets of the first size, and local points of gap grading are produced. Thus particle interference causes an increase of voids, and a corresponding increase in water-cement ratio is needed to maintain consistency. This extra water plus the effect of local gap grading results in marked loss of suspensive power.

Thixotropy.—Thixotropy is characterized by clusters of particles loosely agglomerated such as occur in gap gradings or with particle interference. It is associated by this writer with loss of suspension, with rigidity and rupture, and occurs slowly with time. The writer would expect to find evidence of thixotropic effects in the distortion curves of the new testing machine.

Dilatancy.—The authors employ the terms "rupture" and "dilatancy" synonymously. Structural failure in the case of a ball of stiff paste cited in the paper may result in a separation of the two hemispheres being twisted, without materially increasing the distances between the particles in either hemisphere, or it may be manifested in the segregation of the particles in the plane of shear due to the "troweling action" described which causes the particles to move away from this plane and become closer spaced on either side. Rupture is started by particle interference and produces planar segregation and agglomeration.

The writer believes that dilatancy implies a mutual dilation of all particles in all directions, and not a separation across a plane of shear. He would associate dilatancy with the mechanism of granular structure that maintains plastic suspension.

In the examples of true dilatancy cited in the paper, we see that the effects are produced by acceleration of motion, impulse, or impact. The authors note that deformation of a plastic mass with a high rate of application exhibits a marked dilatancy, "apparently because the particles do not have time to find the paths in which free movement might have taken place."

Applied to concrete mixtures, impact tends to set up instantaneous movement of the matrix in all void pockets and this calls up infinite resistance at all openings, large and small. The simultaneous expansive forces tend to separate the aggregate particles in all directions, and this dilatancy might be termed an acceleration of plastic suspension.

While actual impact would rarely be applied externally to concrete, an effect of impact occurs in the end force of a surge due to wave motion which builds up like the snap of a whip.

Indications of dilatancy in the data with this new machine should appear as a time element on the torque curves.

MESSRS. T. C. POWERS[7] AND E. M. WILER[7] (*authors' closure, by letter*).—As stated in the last paragraph of the paper, we believe the fundamental principles governing the plastic flow of granular suspensions is yet to be established. Therefore, Mr. Weymouth's hypotheses are of interest and will be given full consideration as we search for adequate interpretations of experimental data. It must be said, however, that his hypotheses seem not to take into account certain facts already known.

For example, Mr. Weymouth seems to have overlooked the fact that the forces causing flocculation of the cement particles are responsible for the simultaneous settlement of large and small particles rather than any filtering or "clotting" action such as is pictured by Mr. Weymouth. This is proved by the fact that when particles are dispersed with a suitable agent, segregation of particle sizes does take place. These forces which cause agglomeration are probably strong enough to hold the fine particles rather tightly attached to the coarse ones and thus prevent any "roiling" action such as Mr. Weymouth describes.

The writers cannot agree with Mr. Weymouth's statement that "dilatancy implies a mutual dilation of the particles in all directions. . . ." We believe that the term applies, not to the particles, but to the aggregation of particles and that any part of this aggregation may become dilatant when distortion becomes too great. In the ball-of-cement-paste experiment cited, dilatation occurs first where the unit distortion is greatest.

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[7] Assistant to the Director of Research, and Assistant Physicist, respectively, Portland Cement Assn., Chicago, Ill.

CONCRETE UNDER SUSTAINED WORKING LOADS; EVIDENCE THAT SHRINKAGE DOMINATES TIME YIELD

By G. A. Maney[1]

Synopsis

Evidence is presented to support the conclusion that "plastic flow" or "creep," which have been considered to be the inelastic time yields of concrete under load, are negligible when concrete is loaded under low-stress conditions of ordinary practice. This evidence shows that what has for 30 years been considered an inelastic change due to load is an elastic change due to nonuniform shrinkage. The law of nonuniform shrinkage accounts for most of the excess time yield at ages under one year in loaded over unloaded cylinders which has for so many years been called "creep" or "plastic flow." This paper contends that as long as the *relative* shrinkage differences between elements of a cross section continues, *elastic time yield* of cylinders under sustained load will differ from that of unloaded companion cylinders.

The problem of time yield of concrete under sustained load has for many years been approached on the basis that unit shrinkage in a direction parallel to the axis of a loaded concrete cylinder was uniform over the whole loaded cross-section. Nowhere in the extensive bibliography of "plastic flow" and "shrinkage," prior to 1940, do we find any attempt to determine what the effect on time yield would be, of a much greater shrinkage for the surface than for the interior concrete of a cylinder which dried out when loaded on removal from wet curing.

Those thousands of cylinder test results which have compared surface gage line readings taken parallel to the axis for loaded specimens with the same readings on identical or companion cylinders have all discovered the axial time yield of the loaded cylinder was roughly twice that of the unloaded cylinder. If shrinkage was uniform throughout the mass of a concrete cylinder, this could only mean that a time yield attributable to load intensity and quite independent of shrinkage was taking place. On the other hand, if the shrinkage is not uniform then it may be reasoned that the additional yield under load is a function of shrinkage.

Test Conditions for "Plastic Flow" Measurements

The following series of facts regarding the test conditions should now be mentioned since most of the tests which have been interpreted to make "plastic flow" an important property of concrete under working loads had these conditions in common:

1. Most of these cylinders were loaded on removal from wet or moist curing.

2. The identical or companion cylinder was allowed to dry out in the same relative humidity as the loaded specimens.

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3. The unit load applied to the loaded cylinder was in the range from 300 to 1000 psi. and was of the same average intensity as the design unit loads of practice.

4. The "plastic flow" referred to in this discussion is that which occurs under these low or working stress loadings and is not the "plastic flow" which is universally known to take place at unit load intensities in the region of the elastic limit.

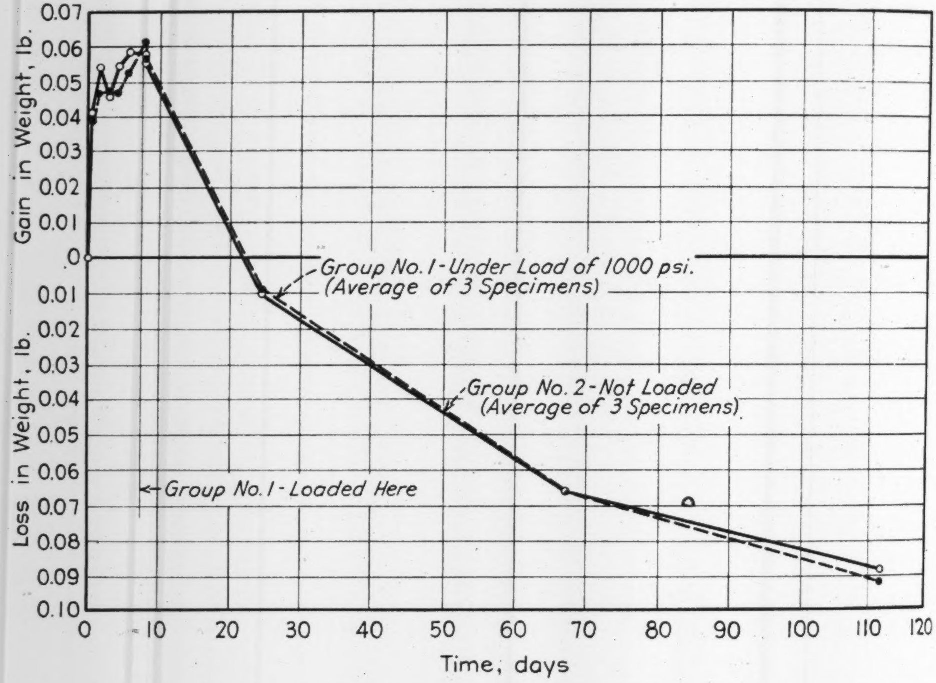

FIG. 1.—A Comparison of Loss of Weight Between Loaded and Unloaded Plain-Concrete Specimens Due to Drying Out of Mixture.

Average weight of specimens = 6.13 lb. Test started at age of 1 day. Group No. 1 placed under continuous load at age of 8 days.

5. The unit time yields were always measured on the surface of the concrete and were based on the average results of three or more gage lines.

WATER LOSS INDEPENDENT OF LOAD INTENSITY

The fact that shrinkage of concrete is related to although not directly proportional to moisture loss or drop in humidity of concrete has long been recognized. It would be reasonable then to expect that if loaded concrete shortened more than unloaded concrete it should perhaps lose more moisture correspondingly and the term "seepage" has crept into the literature of "plastic flow" as a phenomenon which assumes that colloidal moisture is "squeezed" out of loaded concrete in somewhat the same fashion as water might be squeezed out of a sponge. For the loaded specimen this moisture loss is contemplated in addition to the shrinkage water loss which is common to both the loaded specimen and its unloaded companion specimen.

Figure 1 presents a sample of several test results where great care was taken to assure the same surface exposure and same humidity of surrounding air in both loaded and unloaded specimens. These results merely emphasize the fact that moisture loss depends only on the humidity of the surrounding air and is not affected by loading. This evidence indicates that "seepage" does not exist under working load.

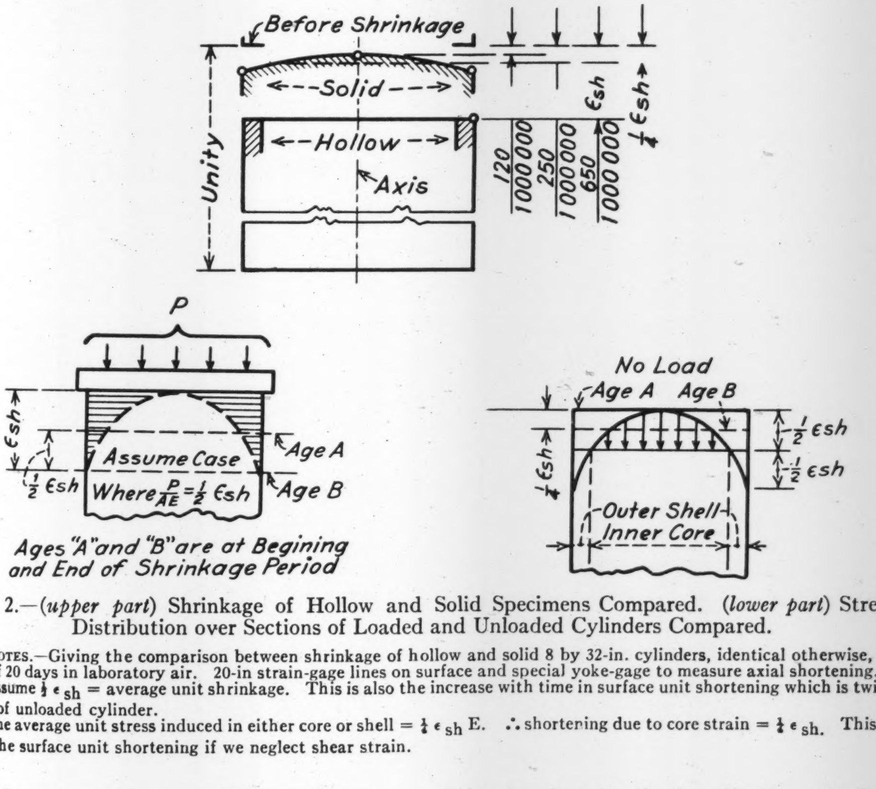

FIG. 2.—(*upper part*) Shrinkage of Hollow and Solid Specimens Compared. (*lower part*) Stress Distribution over Sections of Loaded and Unloaded Cylinders Compared.

NOTES.—Giving the comparison between shrinkage of hollow and solid 8 by 32-in. cylinders, identical otherwise, at age of 20 days in laboratory air. 20-in strain-gage lines on surface and special yoke-gage to measure axial shortening.

Assume $\frac{1}{2}\epsilon_{sh}$ = average unit shrinkage. This is also the increase with time in surface unit shortening which is twice that of unloaded cylinder.

The average unit stress induced in either core or shell = $\frac{1}{4}\epsilon_{sh}E$. ∴ shortening due to core strain = $\frac{1}{4}\epsilon_{sh}$. This is also the surface unit shortening if we neglect shear strain.

NONUNIFORMITY OF WATER LOSS THROUGHOUT VOLUME

An interesting side light on the true distribution of shrinkage in concrete members is found in a paper by Gause and Tucker.[2] This investigation proves what must be correct if "warping" or nonuniform shrinkage occurs in a mass of concrete and that is that surface concrete approaches the humidity of the surrounding air much more rapidly than the interior concrete. In other words, surface concrete dries out more rapidly than interior concrete and it follows directly then that surface concrete shrinks more rapidly than interior concrete.

Nonuniform shrinkage which proceeds rapidly at the surface and very slowly near the longitudinal cylinder axis immediately gives rise to balanced internal stresses in the unloaded or control cylinder such that the total tension induced in the outer ring section is equal to total compression induced in the central axial core. Figure 2 shows the distribution of these induced stresses.

The longitudinal tensile strains induced in the outer ring are decreased by the "Poisson's ratio" effect of the lateral or circumferential stresses induced in this same outer ring. This tendency to decrease the surface gage line readings is largely counteracted by the annular shearing strains set up between the inner compression core and the outer tension ring as one opposes the other. It is reasonably accurate to assume that both these deformation effects are not only small but counteractive. This leads directly to the conclusion that in the unloaded specimen the shrinkage strain recorded by surface gage line readings is closely equal to the induced shortening of the compression core of the cylinder.

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[2] G. Rupert Gause and John Tucker, Jr., "Method of Determining the Moisture Condition in Hardened Concrete," *Journal of Research*, Nat. Bureau Standards, Vol. 25, No. 4, October, 1940, p. 403 (*Research Paper 1334*).

In Fig. 2 we assume a case which is quite typical for a long period after loading and removal from moist storage where shrinkage at center of cross-section is approximately zero and where the shrinkage increase varies according to a parabolic law radially. For the cylinder which is loaded while drying out we see how the increase in deformation as measured by surface gage lines will be one-half of the unrestrained surface shrinkage or of the surface shrinkage tendency. For the unloaded cylinder, this is only one-quarter of the unrestricted surface shrinkage. This relation is verified approximately by the data presented in the upper part of Fig. 2, which shows how the restraint of a nonshrinking inner cylinder core reduces the average shortening as measured by surface gage lines to only about one-quarter of that for a hollow cylinder of identical concrete where the restraint of the nonshrinking inner core is absent.

EVIDENCE THAT "FLOW" IS AN ELASTIC FUNCTION OF SHRINKAGE

The approximate 2:1 ratio between the surface shrinkage of loaded and unloaded identical cylinders as developed in the lower half of Fig. 2 is very important and is verified in general by all the experimental data on this subject, regardless of whether large or small time yields are concerned and holds for specimens loaded while curing continues and swelling takes the place of shrinkage. The following elementary series of deductions leads to the logical conclusion that under working load conditions in concrete any large or important distortion attributable solely to load is questionable:

1. If in one series of tests the measured shrinkage is one-half or one-quarter or one-eighth of that of another series of tests, then the so-called "plastic flow" is in the same ratio.
2. This same statement may be repeated with reference to "swelling" under water and is just as correct in this case as for shrinkage in dry air.
3. Some place between a test series with small shrinkage and one with small swelling we pass through a series which might have zero volume change (zero shrinkage or swelling) when unloaded. The companion loaded specimens in this case would still have about the *same* amount of "plastic flow" which would in this case be zero.
4. Therefore, if we can prevent shrinkage we can prevent "plastic flow."
5. If "plastic flow" were only a function of load it could not be prevented.
6. Therefore, one final conclusion must be that what has been called "plastic flow" all these years is merely a phenomenon of "elastic" deformation resulting from nonuniform shrinkage or "warping."

EXPERIMENTAL EVIDENCE OF WARPING OR NONUNIFORM SHRINKAGE

The evidence that the interior core of a cylinder shrinks very little and that it is thrown into compression by the high shrinkage and induced tension of the outer shell elements is very clear from a test like that indicated in Fig. 2. In Fig. 3 and 4 additional proof is given of the nonuniformity of shrinkage throughout a mass of concrete. Figure 3 gives the "warping" distortions of 6 and 12-in.

concrete cylinders split into four equal quarter cylinders by heavy rubber gasket separators, in comparison with those of corresponding solid specimens.

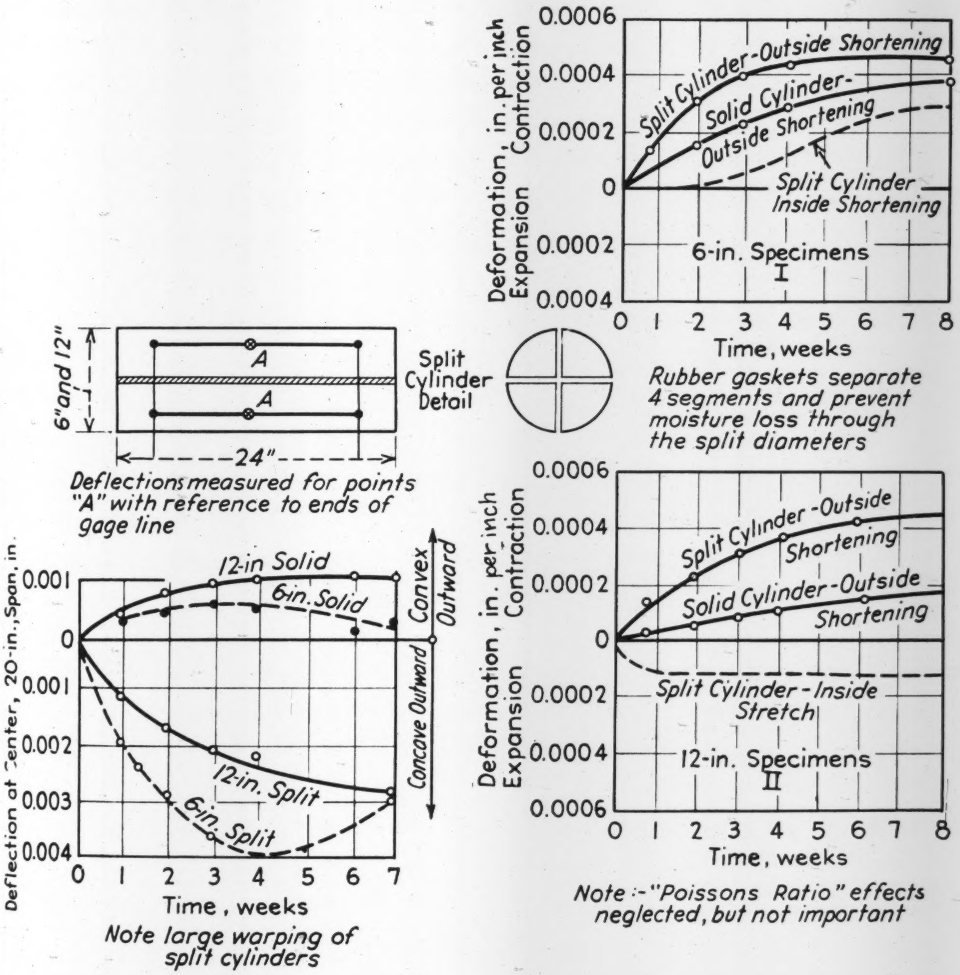


FIG. 3.—A Comparison of Surface Unit Shortening Due to Shrinkage in Unloaded Split and Solid Cylinders, Which Clearly Establishes the Universal Tendency to Warp, Caused by the Tendency of Shrinkage to Increase with Proximity to the Surface.

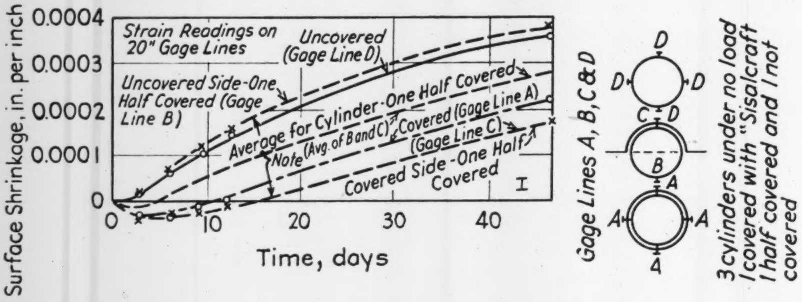
The experimental data illustrates clearly the meaning of "symmetrical" warping. In both "I" and "II" the surface shortening of solid cylinders average that of the split ones. Restraint of surface by core is evident.

The split segments on drying out immediately start to warp in a manner such that the outer surface of the segment is concave outward in a longitudinal direction, due entirely to the more rapid surface shrinkage. Both solid and split cylinders lose the same percentage of moisture since both have the same exposed surface. The rubber gasket prevents moisture loss through the radial crack between the segments. In the solid cylinders, the restraint of this warping is one method of explaining the existence of tension in the outer shell and compression on the inner core of the unloaded or companion specimens.

Figure 4 illustrates another interesting proof of the universal warping tendency of concrete. There three specimens, identical except for obstructions to rapid moisture loss, are compared. The half-covered specimen loses a water percentage close to half way between the loss of the uncovered and the completely covered specimens. The two following results are important:

1. The uncovered specimen has the same average shortening as is measured on the dry side of the half-covered specimen.
2. The half-covered specimen has about the same shortening on the covered side as the average measured on the completely covered specimen.

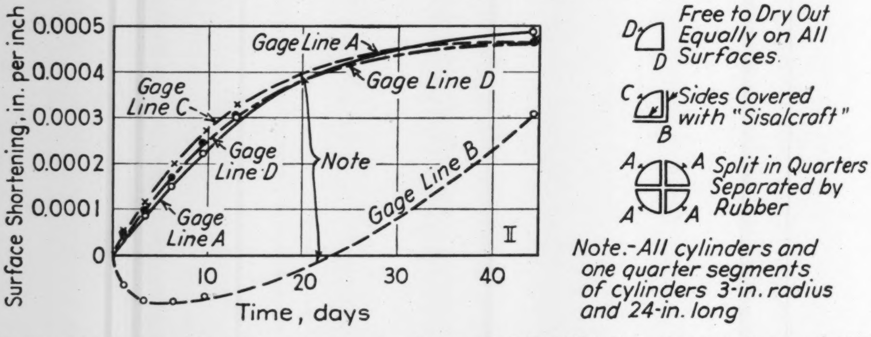
This means that if the half-covered specimens were straightened out to eliminate warping, a compression would exist on the covered side and a tension on the uncovered side. This same condition is brought about in the relation of center compression to surface tension by the restraint of this warping because of symmetrical drying out in the uncovered cylinders.



NOTE 1.—(a) Note how much greater the shortening measured on gage line *B* is than that on the covered side where moisture loss is greatly retarded.

(b) It is seen that the average of shortenings *B* and *C* is about the average of *A* and *D* on the covered and uncovered cylinders.

(c) Note that dry side of one-half covered cylinder shortens much the same as the uncovered cylinder and that the covered side shortens much the same as the average shortening of the all-covered cylinder.



NOTE 2.—Again a comparison of shortenings measured on gage lines *B* and *C* indicate the tremendous warping tendency developed when the tendency to shrink and lose moisture is unsymmetrical.

FIG. 4.—Showing Cylinder Warping Due to Symmetrical Shrinkage.

EXPERIMENTAL EVIDENCE OF THE ELASTIC NATURE OF TIME YIELD

Figure 5 shows a comparison between pairs of hollow and solid cylinders. Each pair consists of a specimen which is alternately loaded and unloaded and another control specimen which remains unloaded. Both specimens dry out under identical conditions. The following conclusions are based on this series:

1. When load is removed from the loaded specimen it fails to return to the initial reading because of the natural increase in the elastic modulus with time. This initial set is not due to sustained load but only to the increased modulus.

2. In the hollow group the time yield tends to be the same for both loaded and unloaded specimens because of absence of restraining center core. In the solid group the typical doubling of the time yield in loaded as compared with the unloaded cylinder is observed.

3. In the loaded specimen of the solid group a recovery is observed after load removal which is just as much an elastic change as the doubled time yield of this specimen. This has been called "plastic recovery," but the common usage of the term "plastic flow" assumes that it is the "*permanent*" change of shape due to load which cannot be recovered when the load is released. All "recovery" must be "elastic" from its very nature.

Concrete columns or plain concrete cylinders with longitudinal compressive

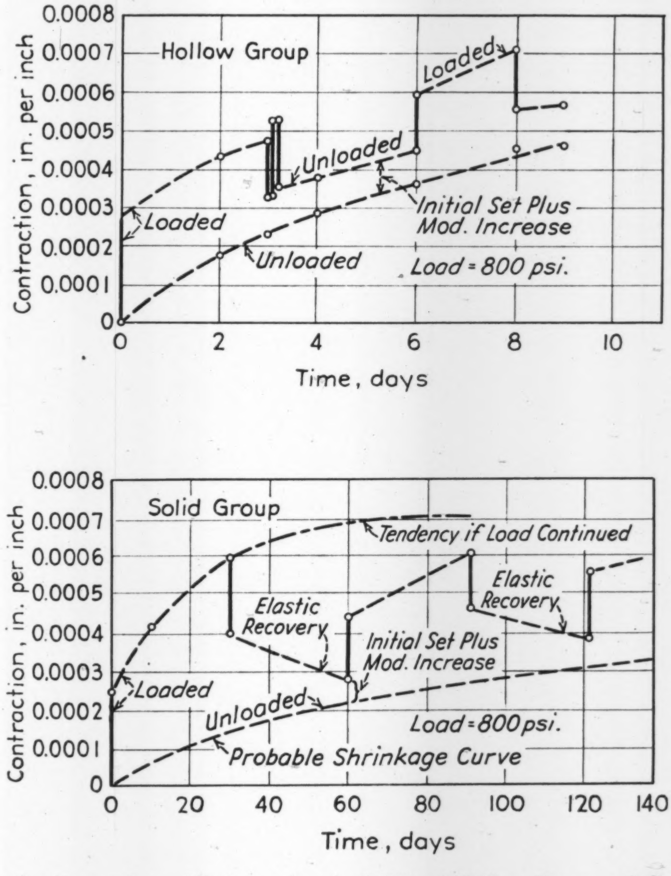


FIG. 5.—Comparison of Deformations in Solid and Hollow Concrete Cylinders.

steel when tested for time yield lead us to similar conclusions regarding the nonimportance of "plastic flow" under working load conditions. Specimens Nos. 24 and 26 of Fig. 6 are reinforced with about 5 per cent of longitudinal steel and specimen No. 22 is unreinforced but of identical concrete. Specimen No. 24 remained unloaded and No. 26 was loaded and unloaded twice with the load held for 18 days with the first loading. All readings on these specimens were taken on reinforcing. Certain results here are typical and have been found in many other tests at Northwestern University. They are listed in the following:

1. The shrinkage of reinforced specimen No. 24 is less than that of unreinforced specimen No. 22 because, although in both the inner core restrains the surface measured shortening, in No. 24 we have the additional restraint due to reinforcing steel which does not shrink at all.

2. The increase in compressive steel stress with time is proportional to the shrinkage of the plain concrete specimen and is not due to plastic flow.

3. On release of load the stress in the steel of specimen No. 26 finally has an elastic recovery to the steel stress of No. 24 which has never been loaded. This is further evidence of "elastic" recovery and of the nonimportance of "plastic flow" under working loads.

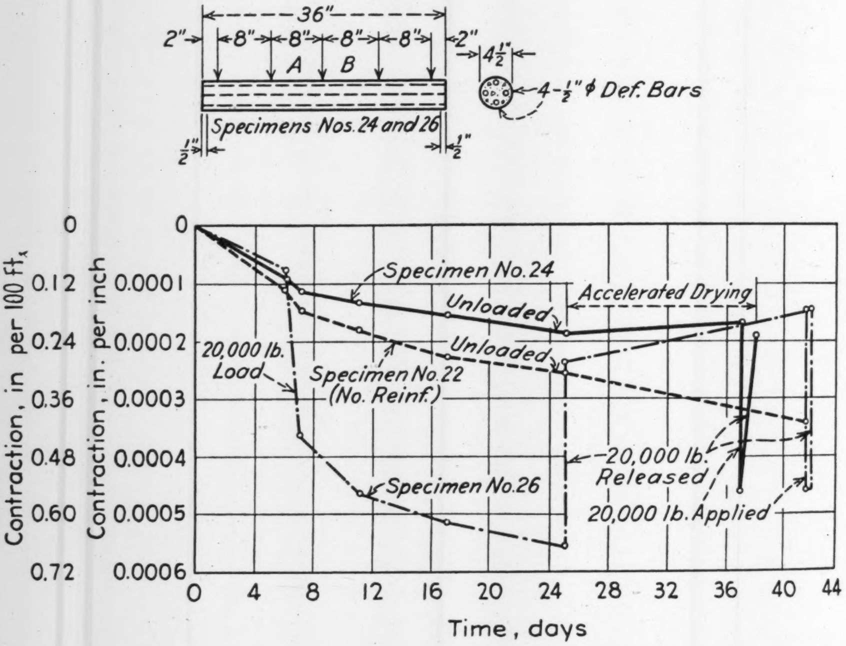


FIG. 6.—Sustained Load Deformations in Longitudinally Reinforced and Plain Concrete Cylinders

QUESTIONS RAISED BY TIME YIELD AT AGES BEYOND ONE YEAR

According to Gause and Tucker[2] and as indicated in Figs. 6, 7, 8, and 9 of their paper, the relative humidity just beneath the surface of concrete specimens of 12-in. minimum dimension reaches its lowest point—surrounding air humidity—in something less than a year. At some age less than a year the maximum difference in humidity between interior and surface of the mass of concrete occurs. Since relative humidity of concrete is a measure of its water loss and tendency toward shrinkage, this elastic differential of increase in loaded over unloaded specimens should reach a maximum in something less than a year's time. Then as the shrinkage of the interior of the mass increases, this differential might be expected to decrease. This tendency is borne out by the reduced warping of the 6-in. split cylinder of Fig. 3 which starts quite early. The lag for the 12-in. specimen is much more. The two questions raised in connection with differential elastic changes at greater ages are:

1. Does the shrinkage tendency of concrete at the interior of the mass, which dries out very slowly, ever approach closely the shrinkage tendency of the surface elements which dry out rapidly? There is circumstantial evidence that it does not. If it did, specimens at ages of 500 days or more should show a tendency of "flow" to decrease.
2. What is the effect of the much greater curing period of the inner core on its modulus of elasticity and how does this affect time yield? Undoubtedly the greater stiffness of the inner core has an effect similar to its reduced shrinkage, and both of these effects tend to keep the stress intensities much higher on the inner core than on the outer shell of cylinders under sustained load.

Much more experimental evidence is needed on the questions raised in this paper, and particularly on changes occurring at greater ages.

CONCLUSION

All the evidence available seems to point to the dominance which shrinkage exerts over all time yields occurring under working load conditions. That some small amount of flow might exist under working loads is not denied, but it is quite evident that all the important time yields under load which have in the past been attributed by all investigators to "plastic flow" are merely elastic redistributions of stress and strain due to nonuniform shrinkage or "warping." It is thought that if much of our tremendous mass of experimental data in this field were re-evaluated on the basis of our new knowledge of nonuniform shrinkage, further support of these conclusions would be made available.

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DISCUSSION

Mr. P. C. Rutledge[1] (*presented in written form*).—Mr. Maney's paper is, I believe, an outstanding contribution to our understanding of the behavior of concrete under working loads for two reasons: (1) he has shown, by a series of careful observations, that time deformations of concrete under sustained working loads, which have been widely interpreted as plastic flow for the past twenty years, may, in fact, be caused entirely by a redistribution of stress resulting from shrinkage; (2) whether or not the evidence proves that time deformations are due exclusively to shrinkage, it does open a new field for speculation and research on concrete.

At the end of his paper Mr. Maney has listed some of the open questions which have not been answered by his results. I should like to point out other possible interpretations of the test evidence.

In the direct evidence presented in this paper none of the tests exceeded eight weeks in duration. In this period the shrinkage curves show a tendency to become horizontal and to leave a residual differential shrinkage between the exterior and interior of solid specimens. This evidence, and results of loss of weight tests over longer periods, do not prove that the centers of solid bodies of concrete will not eventually shrink an amount equal to the exterior shrinkage, with a resulting dissipation of the differential shrinkage stresses. The permanence of differential shrinkage and, in consequence, the validity of the hypothesis that time deformations are caused largely by differential shrinkage, depend entirely on continued differential hydration of the cement in the interior of the concrete. In other words, some of the water in the interior will be lost progressively by chemical combination rather than by evaporation. Circumstantial evidence from these tests, from tests of longer duration, and from studies of long-time hydration of cement indicate that Mr. Maney's conclusions in regard to the permanence of differential shrinkage are correct. I wish to point out that there is no direct evidence to prove this point.

Mr. Maney has assumed that the effect of Poisson's ratio on lateral and longitudinal deformations, annular shear deformations, and lateral shrinkage will counteract each other and, hence, are negligible in their final effect. If longitudinal differential shrinkage occurs, and Mr. Maney has shown this to be a fact for the period of his tests, differential shrinkage in lateral directions must also exist. Therefore, circumferential tensions and radial compressive forces of approximately the same magnitude as the longitudinal shrinkage forces will be developed in cylindrical specimens. Tests by Richart, Brandtzaeg, and Brown[2] show that the modulus of elasticity of concrete subjected to lateral compression in a triaxial compression test decreases to about one half of the modulus resulting from a simple compression test. Furthermore, the decrease in elastic modulus is substantially independent of the magnitude of the lateral compressive stress. Thus, it appears possible that the difference in changes in length between loaded and unloaded specimens can be explained partially by a decrease in the elastic modulus resulting from radial compressive stresses set up by lateral shrinkage. If the magnitude of the longitudinal shrinkage compressive stresses should be approximately equal to the compressive stresses due to applied loads, and if the change in elastic modulus should occur gradually as the shrinkage stresses increase, the effect of lateral shrinkage on the elastic modulus presents another complete explanation for the difference in shortening of unloaded and loaded specimens in long-time tests.

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[1] Associate Professor of Civil Engineering, Purdue University, Lafayette, Ind.

[2] F. E. Richart, A. Brandtzaeg, and R. L. Brown, "A Study of the Failure of Concrete Under Combined Compressive Stresses," *Bulletin No. 185*, Engineering Experiment Station, University of Illinois, November, 1928.

Neither the shrinkage phenomena discussed by Mr. Maney nor the effect of possible changes in elastic modulus is directly related to plastic flow, although the action under the latter explanation is more similar to a plastic action. It is interesting to note that the tests of Richart, Brandtzaeg, and Brown on concrete both in triaxial compression and in spirally reinforced columns, and tests which we have carried out at Purdue on dense sand and gravel mixtures in triaxial compression, show that, as the lateral confining forces acting on these materials increase, they become much less brittle and behave more like plastic materials. Failure under these conditions occurs only after relatively large deformations. Many of the specimens show the characteristic lateral bulging failure typical of plastic materials. These observations are a natural consequence of the decrease in initial modulus of elasticity for laterally confined concrete.

The object of this discussion is to support Mr. Maney's contention that a large amount of experimental and investigational work still can be done on the physical properties of concrete. The conditions of shrinkage of concrete specimens and the initial tensile and compressive stresses at the time they are tested will have a decided effect on the test results. The effects of these conditions should be considered carefully in the simple compression tests and beam tests which are widely used today. It is easily possible that the results of tests on concrete cylinders which have never been allowed to dry, or tests on cylinders which, arbitrarily, have been allowed to dry in laboratory air 24 or 48 hr. before test, will be quite different from the actual behavior of the concrete as it is used in the field. By perspicacious observation on a few relatively simple tests, Mr. Maney has demonstrated convincingly how little we know about the material which has been studied for many years.

MR. GERALD PICKETT[3] (*presented in written form*).—The paper by Mr. Maney shows that he has given a very difficult problem considerable thought. An analysis of stress and strain in finite bodies undergoing nonuniform volume change is a problem that has puzzled the best of mathematicians and elasticians. However, by using the energy methods developed by Timoshenko and Ritz,[4] certain of these theoretical problems can be solved to any desired degree of accuracy.

For example, the writer has made an analysis, neglecting the effect of Poisson's ratio, for the stresses, strains and displacements of an elastic prism drying from two opposite sides.

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[3] Research Physicist, Portland Cement Assn., Chicago, Ill.

[4] S. Timoshenko, "Theory of Elasticity," Article 43, p. 151 and Article 65, p. 203, McGraw-Hill Book Co., Inc., New York, N. Y.

Walter Ritz, "Gesamelte Werke," pp. 192–264, published for Société Suisse de Physique, by Gauthier-Villars, Paris (1911).

Some results for two ratios of length to thickness are given in the accompanying Fig. 1.

The results show that if the length-to-thickness ratio is at least ten, then planes a short distance away from the end regions remain essentially plane. However, some warping occurs in the end regions, and the dimensions of most concrete specimens are such that a large part of the specimen is in the region of end effect and, therefore, careful measurements of over-all change of length taken upon the surface and upon the longitudinal axis should not agree.

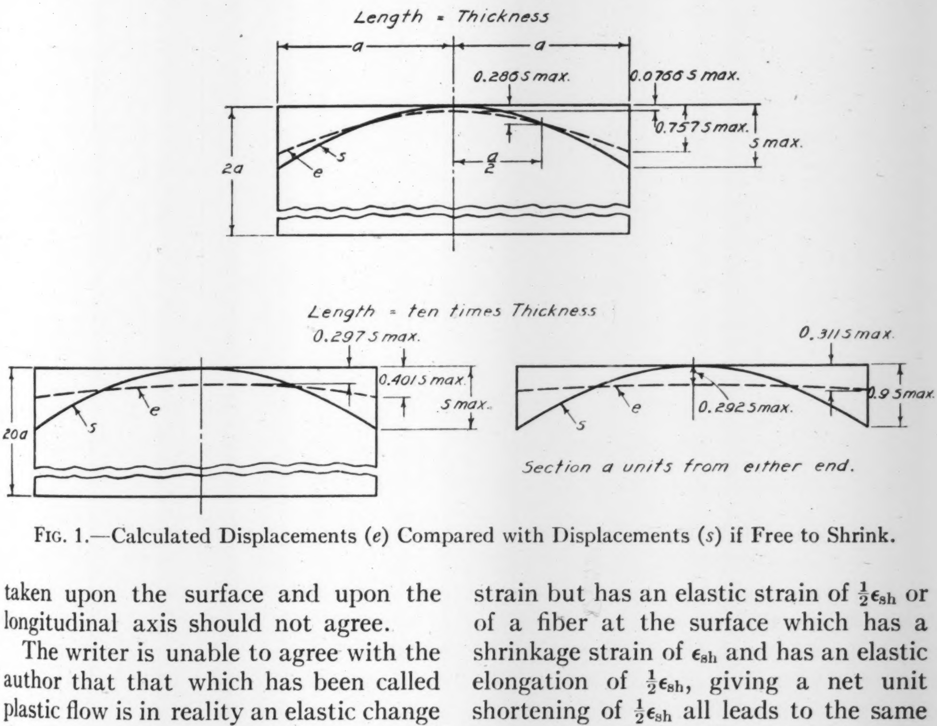

FIG. 1.—Calculated Displacements (e) Compared with Displacements (s) if Free to Shrink.

The writer is unable to agree with the author that that which has been called plastic flow is in reality an elastic change due to nonuniform shrinkage. The analysis shown in the author's Fig. 2 is an important factor in his argument. Assuming concrete to be elastic, he has shown that when a cylinder is under load the average shortening caused by shrinkage is $\frac{1}{2}\epsilon_{sh}$. This is illustrated by the distance between lines marked age A and age B.

This analysis is correct, but his analysis of the unloaded cylinder is incorrect. He has taken the average elastic strain in the core, $\frac{1}{4}\epsilon_{sh}$, to be the unit shortening of the core and has omitted the average shrinkage-strain of the core which is also $\frac{1}{4}\epsilon_{sh}$. The total strain in the core is therefore $\frac{1}{2}\epsilon_{sh}$. The value of $\frac{1}{2}\epsilon_{sh}$ may also be found by considering a fiber at the junction of the core and shell. Such a fiber does not have elastic strain but has shrinkage strain equal to $\frac{1}{2}\epsilon_{sh}$. Consideration of a fiber at the center which does not have shrinkage strain but has an elastic strain of $\frac{1}{2}\epsilon_{sh}$ or of a fiber at the surface which has a shrinkage strain of ϵ_{sh} and has an elastic elongation of $\frac{1}{2}\epsilon_{sh}$, giving a net unit shortening of $\frac{1}{2}\epsilon_{sh}$ all leads to the same conclusion. This tedious analysis could be avoided by applying the principle of superposition. For elastic materials this principle requires that shrinkage effects and load effects be entirely independent, which means that shrinkage and load could not interact to cause additional deformation. Therefore, the effect of shrinkage upon creep cannot be explained as elastic deformation.

The data on solid and hollow cylinders presented by Mr. Maney are irrelevant, because it is not logical to assume that a hollow cylindrical shell and the corresponding shell of a solid cylinder, if unrestrained, would shrink the same amount in 20 days because the latter shell would be supplied with moisture from the interior.

The writer agrees with the author that shrinkage and not seepage is the major cause of time yield under load, and the above statements are not intended to detract from this important contribution of the author.

The writer, in unpublished work, has shown that the hypothesis that the stress-creep coefficient increases rapidly as the ultimate strength is approached is sufficient to account for the effect of shrinkage upon time yield.

MR. C. A. MENZEL.[5]—My experience differs a bit from that cited by Mr. Maney. I understand from what he said that if concrete is put under sustained load for some time, water is not squeezed out of it. About two years ago I had occasion to make a long-time pull-out bond test involving two specimens. One specimen was moist cured for seven days and then immediately coated with a thick layer of paraffin in order to reduce moisture loss. In this condition the bar was subjected to pull at one end for a period of something like ninety days. Several days after the load was applied, little bubbles developed under the paraffin at the loaded end of the specimen, and as time went on, more bubbles appeared farther up, but there were no bubbles near the top of the specimen where there was no stress in the concrete. To prove that there was moisture in these bubbles, we pricked them, and concluded that the moisture observed must have been squeezed out of the concrete approximately in proportion to the compressive stress developed.

The second companion specimen was cured continuously in the moist room for about ninety days and then tested along with the first specimen. It developed a lower ultimate load than the one that had been continually exposed to high load from the age of seven days. It seems that creep or flow under the continued loading had a favorable effect.

Though my experience in long-time tests has been very meager, it seems strange to me, in view of Mr. Maney's experience, that the only continuously loaded specimen I ever had under observation definitely showed that moisture was squeezed out of it.

MR. R. E. DAVIS.[6]—Just because a compression specimen loses no weight, I do not believe that it should be concluded that water may not have been squeezed from the gel structure. Concrete contains many tiny voids not occupied by gel. Unless the concrete is kept completely saturated, which could only be the case with a very small specimen, these void spaces which were originally filled with water become partially evacuated as hydration proceeds and as a portion of the free water in these voids is adsorbed by the gel. That such action takes place has been demonstrated experimentally, where negative pore pressures as large as −12 psi. have been observed in unrestrained sealed concrete specimens maintained at constant temperature. If to such specimens continuous load is applied, a portion of the water of adsorption may be slowly forced from the gel again to become free water in the pores and the negative pore pressure may be partially relieved.

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[5] Associate Engineer, Research Laboratory, Portland Cement Assn., Chicago, Ill.

[6] Professor of Civil Engineering, and Director, Engineering Materials Laboratory, University of California, Berkeley, Calif.

Under these conditions plastic flow takes place without change in weight.

Specimens sealed in copper jackets and not loaded until they were of considerable age have been observed gradually to change in length after such loading, increasing for a long period of time in length if in tension, and decreasing in length if in compression.

In the interior of dams, where there could have been no change in moisture content over the period of observation, it has been noted that there are long and steady changes in length as determined by strain gage and strain meter measurements.

Not so long ago, I was called to examine a cold-storage warehouse in California—flat slab construction columns about 20 ft. on centers—built over twenty years ago. Here the floors have been subjected to more or less constant conditions as regards temperature, humidity, and load for many years. Under these conditions the slabs and exterior columns have been gradually deflecting, until now, in the middle of the panels, as measured on the diagonal, there are places where the deflection is as much as 3 in., and in some places the deflection of the reverse curve in the exterior columns is as much as an inch.

I should like to have Mr. Maney account for these changes which I have just described on the basis of elastic deformations.

MR. G. A. MANEY[7] (*author's closure, by letter*).—The principal contention of the writer's synopsis seems to have weathered the storm of criticism which any paper like this one, that attempts to present evidence upsetting the conclusions of a thirty-year accumulation of experimental investigation, will inevitably arouse. This contention will be restated briefly by quoting the third sentence of the synopsis:

"The law of nonuniform shrinkage accounts for most of the *excess* time yield at ages under one year in loaded *over* unloaded cylinders which has for so many years been called 'creep' or 'plastic flow'."

As Mr. Rutledge has pointed out in his excellent and helpful discussion, this investigation is not a long-time investigation, but he agrees that a differential of shrinkage would tend to be maintained indefinitely because of the much greater insulation against water loss and the longer curing of the inner core as compared with the outer shell of the cylinders. However, thousands of tests recorded in many leading laboratories have proved that practically all of what has been called "flow" or "creep" has occurred before the shrinkage curve first approaches an asymtotic state with reference to the time axis. The evidence of these tests definitely covers this period.

Mr. Rutledge, in addition to pointing out the need for further investigation, has also pointed out an interesting parallelism between these tests and those of Richart, Brantzaeg and Brown on triaxially loaded concrete, pointing out that the reduction in elastic modulus of triaxially over uniaxially loaded concrete should show up in drying out cylinders under progressive differential shrinkage. Unpublished data from our laboratory and much published data proves that this is so and that the elastic modulus becomes progressively less than that of moist control specimens as this differential shrinkage increases.

The writer appreciates the acknowledgement in the excellent discussion of his severest critic, Mr. Pickett, that "shrinkage and not seepage is the major cause of time yield under load." This is, as has been mentioned, the principal message of the evidence of the paper under discussion. Mr. Menzel, however, does not seem to agree to this conclusion. The phenomenon which Mr. Menzel observes is known to the author, but in his opinion does not in any way contradict the conclusions of Fig. 1 of the paper discussed. If Mr. Menzel should ever get an opportunity to weigh these "bubbles" of water which seem to be squeezed out of a loaded covered specimen he would doubtless be shocked at the insignificant value of ratio of the weight of these water bubbles to the total weight of moisture *drying* out of both loaded and unloaded specimens.

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[7] Chairman, Department of Civil Engineering, Northwestern Technological Institute, Northwestern University, Evanston, Ill.

Mr. Pickett makes two basic statements in his well-considered discussion upon which most of his criticism is founded, which the writer considers erroneous. The first refers to the lower part of Fig. 2 and is as follows, in part:

"This tedious analysis could be avoided by applying the principle of superposition. For elastic materials this principle requires that shrinkage effects and load effects be entirely independent, which means that shrinkage and load could not interact to cause additional deformation."

The author denies the validity of the principle of superposition in this case, even though admitting his analysis to be approximate but not necessarily tedious. The commonest structural application of the principal of superposition is in the construction of influence lines for long span bridges. The simple reason that the principle is here denied is that reason which makes influence lines correct for truss bridges or arches but incorrect for suspension bridges. Briefly, the law of superposition neglects the effect of changes in shape of structure under conditions of actual loading. This change of shape cannot be neglected in the case of a cylinder which shrinks triaxially in addition to developing certain cracking tendencies in the exterior shell. Furthermore, the modulus of elasticity develops a variation throughout the cross-section, being higher in the center core. The author can envision no structure less suitable to rigid mathematical analysis or to the law of superposition than the concrete cylinder in question.

The other statement by Mr. Pickett that demands consideration refers to the upper part of Fig. 5 and the upper part of Fig. 2 and reads as follows:

"The data on solid and hollow cylinders presented by Mr. Maney are irrelevant, because it is not logical to assume that a hollow cylindrical shell and the corresponding shell of a solid cylinder, if unrestrained, would shrink the same amount in 20 days because the latter shell would be supplied with moisture from the interior."

The author had some doubts along this line himself until he saw the paper by Gause and Tucker[8] which shows how tremendously more rapid is the drying out (and shrinkage) of elements close to the surface compared with that of core elements. He had every reason to conclude that the outside surface of the solid cylinder dried out at much the same rate as that of the hollow cylinder. It would be only a slight overstatement of Mr. Pickett's contention to say that shrinkage effects must be almost uniform throughout a solid cylinder. Figures 3 and 4 present ample evidence that this is anything but a correct statement. The hollow-solid cylinder comparison of the upper part of Fig. 5 also presents evidence to disprove the contention of Mr. Pickett that the shrinkage effect is not elastic. In connection with Fig. 5 which presents the most important supporting data, and the lower part of which is a rearrangement of data from previously published results, the author wishes to draw two conclusions and raise two questions for discussion at some future opportunity.

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[8] G. Rupert Gause and John Tucker, Jr., "Method of Determining the Moisture Condition in Hardened Concrete," *Journal of Research*, Nat. Bureau of Standards, Vol. 25, No. 4, October, 1940, p. 403 (*Research Paper 1334*).

Conclusion 1.—In the *solid* specimen the time increase with loading proceeds at about *twice* the rate of the companion solid unloaded specimen, whereas the *same* rate of increase applies to both loaded and unloaded *hollow* specimens. From this we must conclude that when shrinkage is uniform throughout the section as is practically the case in the hollow specimens, the total time yield is the *same* on both loaded and unloaded specimens. Also where shrinkage is nonuniform as in the case of the solid specimens the total time yield is about *twice* as much on the loaded as on the unloaded specimen. This increment of increase of the loaded over the unloaded solid specimen has been what is referred to as "creep" or "flow" in most of our literature on the subject.

Question 1.—If the specimens with uniform shrinkage *have no* "time yield" and specimens with nonuniform shrinkage *do have* "time yield," is it not reasonable to conclude that nonuniform shrinkage is the cause of so-called "flow," which has long been found to be about the same as the shrinkage yield, regardless of age or of unit stress intensity? As a corollary to this question, how can a time yield independent of unit load intensity be called either "flow" or "creep"?

Conclusion 2.—The upper part of Fig. 5 indicates that most of what little time yield could be discovered was due to initial set and increase of elastic modulus with time. In the lower part of the figure the loaded specimen has an *elastic* recovery which fails to return to shrinkage shortening of the unloaded specimen *only* by this same yield due to initial set and increased modulus. In neither of these cases is the *permanent* change of shape or "flow" found to be a function of sustained load.

Question 2.—How then can the time yield be anything except an *elastic* phenomenon? Also, how can a *recovery* be *plastic*, by very definition of the term plastic flow, which means *permanent* change of shape?

The questions raised by Mr. Davis are interesting but are beyond the scope of this paper, since they are concerned with long-time phenomena.

This paper stresses for the first time the importance of nonuniform shrinkage and proposes to establish the fact that what has been called "flow" or "creep" for solid loaded specimens during their first drying out period under working stresses, is practically all due to nonuniform shrinkage. This leads to the important conclusion that under working loads "shrinkage dominates time yield." In conclusion, there is no attempt to deny the existence of flow, particularly where unit stresses are high. The author definitely feels however that under working stress conditions flow is unimportant compared with shrinkage.

THE EFFECT OF CAPPING METHODS AND END CONDITIONS BEFORE CAPPING UPON THE COMPRESSIVE STRENGTH OF CONCRETE TEST CYLINDERS

BY G. E. TROXELL[1]

SYNOPSIS

Compression tests have been made on 768 cylinders of carefully controlled concrete to determine the effect of various types of capping materials upon the observed compressive strength of the cylinders. The capping methods and materials used included: (1) Hydrostone, a high-strength gypsum product; (2) Castite, a sulfur-silica mixture; (3) oiled steel shot; (4) dry steel shot; (5) plaster of Paris tested at age of ½ hr.; and (6) plaster of Paris tested at age of 1 hr.

As the end conditions of the concrete cylinders before capping were likely to affect the results obtained, four definite end conditions were selected for investigation. These included (1) plane ends normal to the axis of the cylinder, (2) plane ends not normal to the axis, (3) convex ends, and (4) concave ends. Tests were made on cylinders of medium- and high-strength concrete.

The quality of concrete is ordinarily judged by the compressive strength of cylindrical test specimens. However, the observed strength of a standard moist-cured specimen is affected not only by the quality of the concrete but also by such factors as method of capping and kind of capping material; end conditions of the uncapped cylinder; end conditions of the capped cylinder; thickness of cap; eccentricity of loading; and rate of load application. This paper is concerned only with the first two factors.

Concrete cylinders cast in the laboratory will usually have a more or less perfect lower end due to the mold being secured to a machined base plate. Furthermore, the upper end is usually reasonably smooth and normal to the axis of the specimen. In contrast thereto, specimens cast in the field often have concave or convex bottom ends, due perhaps to their being cast in molds with flexible tin bottoms, and their top ends are commonly rough, nonplanar and not normal to the axis of the cylinder. One object of the present investigation was to determine the influence, if any, of such conditions upon the compressive strength of the cylinders, even though they be capped to produce plane, parallel ends.

Various methods and materials have been used for capping the irregular ends of concrete cylinders. Plaster of Paris has been used quite commonly, but as it has a low strength (about 1700 psi. at 1 hr. when tested in the form of 2 by 4-in. cylinders) and tends to flow under load, many laboratories have abandoned its use. Neat cement or cement mortar caps are used in various laboratories, but even though they possess high strength, they have certain undesirable characteristics. Their slow rate of hardening requires that the cylinders be capped several days before testing and their high shrinkage tends to produce uneven, checked caps having poor bond to the specimen, especially for large cylinders. In addition to cement, two other high-strength capping materials have more recently come into use. One of these is a gypsum compound known as Hydrostone. It will develop a compressive strength of about 5000 psi. within 1 hr. when tested in the form of 2 by 4-in. cylinders. The other is a sulfur-silica mixture, applied in a molten condition, which will develop a strength of about 6000 psi. as soon as it cools and 8500 psi. within one day. The shot cap method, using small steel shot retained in a metal capping head, with shot fitting into irregularities in the ends of the specimens, has been employed by a few laboratories. Some laboratories have ground the irregular ends to a plane normal surface but ordinarily the cost is prohibitive even for small mortar cylinders.

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[1] Associate Professor of Civil Engineering, University of California, Berkeley, Calif.

Various methods and materials for capping test cylinders were included in this investigation to determine their effect upon the observed compressive strength of identical concrete specimens.

CONCRETE SPECIMENS

All concrete specimens were 3 by 6-in. cylinders having a maximum aggregate size of $\frac{3}{4}$ in. They were cast in two series having nominal compressive strengths of 3000 and 8000 psi., respectively. Before capping, all specimens of each group had one of the four following end conditions: (1) plane ends normal to the axis of the cylinder; (2) plane beveled ends having a slope to the normal of $\frac{3}{16}$ in. in the 3-in. diameter of the cylinder; (3) convex ends having a bulge of $\frac{3}{16}$ in.; and (4) concave ends having a concavity of $\frac{3}{16}$ in. The two ends of each concrete cylinder were shaped alike. The different end conditions were obtained by inserting circular steel parts having the required variations in thickness, in the bottom and the top of the machined cast iron molds.

Twenty-four specimens were made from each batch of concrete—six specimens of each end condition so that for each batch of cylinders one of each of the six methods of capping selected for test could be applied to each of the four types of cylinder ends. Sixteen batches were made for each of the two grades of concrete, giving a total of 768 cylinders, sufficient for 16 independent tests of each variable involved.

TABLE I.—PROPERTIES OF CAPPING MATERIALS.

Material	Compressive Strength, psi.			Modulus of Elasticity at 1 hr., psi.[a]
	Age ½ hr.	Age 1 hr.	Age 6 hr.	
Plaster of Paris	1650	1700	1700	540 000
Hydrostone	4700	5000	5200	1 580 000
Castite	5500	5800	6700	2 150 000

[a] Secant modulus at 1000 psi.

CAPPING MATERIALS AND METHODS

Both ends of each cylinder were capped using one of the following materials: plaster of Paris; Hydrostone; Castite, a molten mixture of sulfur and finely divided silica (similar to Leadite); and $\frac{1}{16}$-in. steel shot with a suitable retaining head. Steel shot in both dry and oiled conditions were used. The compressive strength and modulus of elasticity of the first three capping materials were determined from tests of 2 by 4-in. cylinders. The test results are shown in Table I.

The plaster-of-Paris, Hydrostone, and Castite caps were $\frac{1}{16}$ in. thick at the thinnest portion and $\frac{1}{4}$ in. at the thickest portion. The shot caps were about $\frac{3}{16}$ in. thicker all around.

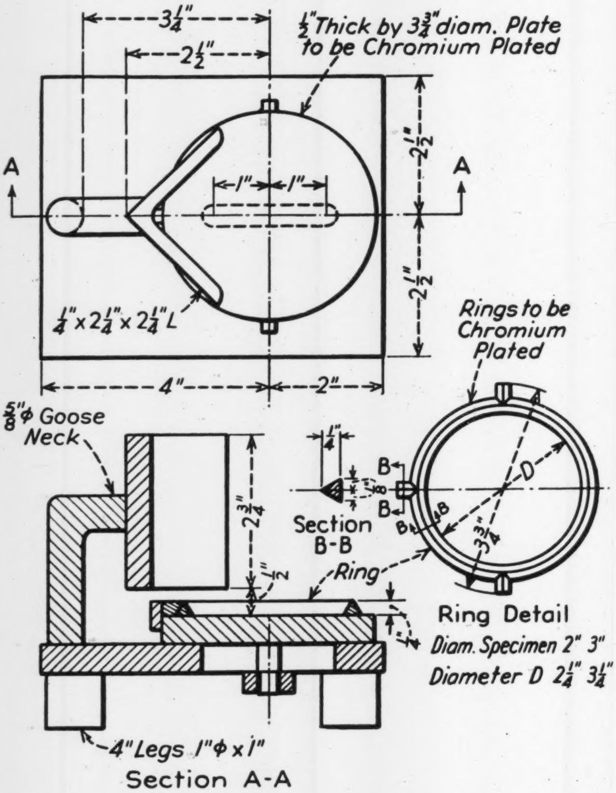
One set of concrete specimens having

caps of plaster of Paris was tested as soon as the caps had hardened for ½ hr. in ordinary air. A second set was tested when the caps were 1 hr. old after standing for ½ hr. in ordinary air and then ½ hr. under a damp sack to prevent drying of the concrete. The Hydrostone caps were treated similarly to the second set of plaster-of-Paris caps and were 1 hr. old when tested. The Castite caps were from one to several days old when tested, as after the first day the gain in strength is small. Furthermore, as Castite is unaffected by moisture, it can be used to cap large numbers of cylinders at one time, the cylinders then being placed in moist storage for long periods before testing.

The relative costs (considering materials and labor) of the various methods of capping cylinders may be briefly summarized as follows:

1. Hydrostone is the most expensive material; also the labor cost is relatively high due to the time required between applications of the top and bottom caps and the usual inability to employ this time effectively for other duties. The over-all cost of Hydrostone caps is high.

2. Castite is high in initial cost but it has a high salvage value because it may be remelted and used about four to eight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Ring
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Diameter D 2¼" 3¼"
4" Legs 1" ϕ x 1"
Section A-A

FIG. 1.—Sulfur Capping Apparatus.

Adjustable for 2 to 3-in. diameter cylinders. Similar design used for larger sizes. Small sizes usually constructed with three units per gang.

times. For fairly regular ends, this method, using the apparatus shown in Fig. 1, is speedy and has the lowest overall cost, especially when the Castite is melted in a controlled oven to eliminate overheating and any need for stirring.

TABLE II.—EFFECT OF CAPPING MATERIALS AND END CONDITIONS BEFORE CAPPING UPON THE COMPRESSIVE STRENGTH OF CONCRETE CYLINDERS, PSI.

Type of Cap	Series I, 3000-lb. Concrete				Series II, 8000-lb. Concrete			
	Plane Normal Ends	Plane Beveled Ends	Convex Ends	Concave Ends	Plane Normal Ends	Plane Beveled Ends	Convex Ends	Concave Ends
Hydrostone	2920	2900	2970	2880	8610	8100	8360	7950
Castite	2820	2860	2750	2940	8780	8700	8200	7700
Oiled shot	2570	2640	2710	2190	7670	7750	7760	4800
Dry shot	2570	2690	2750	2140	7560	7400	7680	4690
Plaster of Paris at ½ hr.	2790	2510	2620	2530	7360	5840	4680	2960
Plaster of Paris at 1 hr.	2830	2470	2570	2560	7470	5670	4640	2930

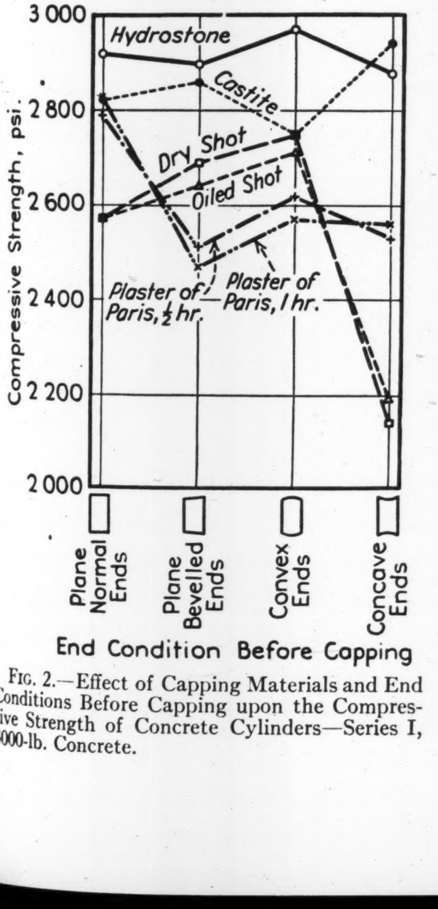

FIG. 2.—Effect of Capping Materials and End Conditions Before Capping upon the Compressive Strength of Concrete Cylinders—Series I, 3000-lb. Concrete.

3. Shot caps cost practically nothing for materials, but the cost of cleaning the shot, especially when oiled, partly offsets this feature.

TABLE III.—RELATIVE COMPRESSIVE STRENGTHS OF CONCRETE CYLINDERS FOR VARIOUS CAPPING MATERIALS AND END CONDITIONS BEFORE CAPPING.

(Cylinders with plane normal ends and Hydrostone caps taken as 100)

Type of Cap	Series I, 3000-lb. Concrete				Series II, 8000-lb. Concrete			
	Plane Normal Ends	Plane Beveled Ends	Convex Ends	Concave Ends	Plane Normal Ends	Plane Beveled Ends	Convex Ends	Concave Ends
Hydrostone	100.0	99.3	101.7	98.6	100.0	94.1	97.1	93.1
Castite	96.6	97.9	94.2	100.7	102.0	101.0	96.2	89.4
Oiled shot	88.0	90.4	92.8	75.0	89.0	90.0	90.1	55.7
Dry shot	88.0	92.1	94.2	73.3	87.8	86.0	89.2	54.5
Plaster of Paris at ½ hr.	95.5	86.0	98.7	86.6	85.5	67.8	54.4	34.4
Plaster of Paris at 1 hr.	96.9	84.6	88.0	87.7	86.8	65.9	53.9	34.0

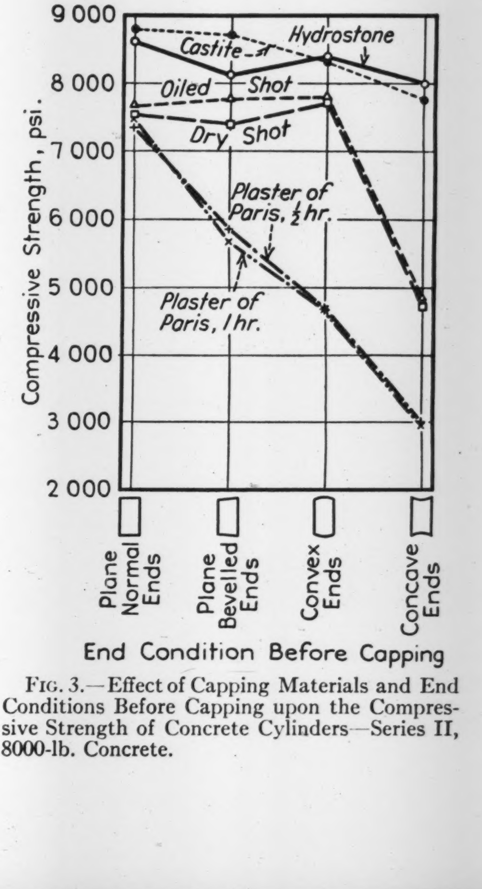

FIG. 3.—Effect of Capping Materials and End Conditions Before Capping upon the Compressive Strength of Concrete Cylinders—Series II, 8000-lb. Concrete.

4. Plaster of paris is inexpensive, but the cost of applying is the same as for Hydrostone and greater than for Castite caps.

COMPRESSION TESTS AND RESULTS

All cylinders were stored in moist air, except for $\frac{1}{2}$ hr. after capping, and then were tested in compression at the scheduled age of the specimen and its caps. A uniform machine idling speed of 0.06 in. per min. was employed for testing all specimens.

The results of the tests are presented in Tables II and III, and in Figs. 2 and 3. From a study of these results the following facts concerning each of the capping methods appear significant:

TABLE IV.—MAXIMUM DIFFERENCE BETWEEN THE COMPRESSIVE STRENGTH OF SPECIMENS WITH IRREGULAR ENDS AND THAT OF CORRESPONDING SPECIMENS WITH PLANE NORMAL ENDS, PER CENT.[a]

Type of Cap	Series I, 3000-lb. Concrete		Series II, 8000-lb. Concrete	
	Over	Under	Over	Under
Hydrostone	+1.7	−1.4	0	−7.7
Castite	+4.2	−2.5	0	−12.3
Oiled shot	+5.4	−14.8	+1.2	−37.4
Dry shot	+7.0	−16.7	+1.6	−38.0
Plaster of Paris at ½ hr.	0	−10.0	0	−59.7
Plaster of Paris at 1 hr.	0	−12.7	0	−60.8

[a] Based upon average strengths of the 16 specimens in each group having identical caps and end conditions.

Hydrostone caps developed high and uniform strengths for both series of specimens. For series I, 3000-lb. concrete, the strengths were higher than for any other material, and for series II, 8000-lb. concrete, the strengths were a close second to the highest values. The various end conditions appeared to have less effect upon the strengths of cylinders capped with Hydrostone than for any of the other materials used. As shown in Table IV, the maximum difference between the compressive strength of specimens with irregular ends and the strengths of corresponding specimens with plane normal ends was only 1.7 per cent in series I and 7.7 per cent in series II.

Castite caps developed high strengths for both series of specimens, especially those of series II, and for all end conditions. However, the variations in strength due to the various end conditions were greater than for the Hydrostone caps but less than for shot or plaster-of-Paris caps. As shown in Table IV, the maximum difference between the compressive strength of specimens with irregular ends and the strength of corresponding specimens with plane normal ends was 4.2 per cent in series I and 12.3 per cent in series II. It is of interest that the specimens having Castite caps in series I were the only ones of either series which showed a higher strength for concave ends than for plane ends.

Shot caps, in general, developed strengths which were intermediate to the high strengths resulting from the use of Hydrostone or Castite caps and the low strengths resulting from the use of caps of plaster of Paris, although in series I the cylinders with plane and concave ends capped with shot definitely gave the lowest strengths for these end conditions, considering all capping materials. As shown by Table IV, and by Figs. 2 and 3, the effect of irregular end conditions for series I was more marked for shot caps than for any other type of cap. For series II, the effect of irregular end conditions on specimens having shot caps was intermediate to that for the high strength Hydrostone and Castite caps, and the low strength plaster-of-Paris caps. The cylinders with convex ends capped with shot showed higher strengths than for the cylinders with plane ends, especially in series I, while the cylinders with concave ends of both series showed decidedly lower strengths than the cylinders with plane ends. This was evidently due to the radial components of the forces produced by the shot acting on the convex and concave ends. In general, the difference in results obtained by the use of oiled as compared with dry shot does not warrant the extra work involved in using oiled shot.

Plaster-of-Paris caps on cylinders of normal strength concrete having plane ends normal to the axis of the cylinder gave strengths comparable to those obtained when using the high-strength capping materials, but they gave very low strengths when the ends were not of this type, as shown by Table IV and by Figs. 2 and 3. The irregular end conditions of high-strength cylinders had a greater effect in lowering the strength when capped with plaster of Paris than for any other capping material. In general, caps of plaster of Paris tested at ½ hr. gave slightly higher strengths than caps tested at 1 hr., due possibly to slight absorption of moisture by the 1-hr. caps which may have occurred while standing ½ hr. under a damp sack to prevent drying of the concrete; but the differences were not appreciable.

Although most specimens developed the common cone fracture near their mid-length, some of the capping materials and end conditions caused special characteristic fractures of the concrete cylinders which indicated how such factors had an effect upon the compressive strength. All shot-capped cylinders with concave ends tended to split into columnar elements due to radial tension. The cylinders having plaster-of-Paris caps on concave ends tended to rupture near their top and bottom rims along planes at about 45 deg. with the axis of the cylinder, due possibly to a combination of radial tension and axial compression; those having convex ends tended to split into columnar elements, due possibly to yielding of the thicker bed of plaster near the edge of the cap so that it did not produce the customary friction on the end surface.

To show the general uniformity of the test results for the individual specimens and the relative reliability of the average strengths reported, the average variations of the individual test values from the mean value for each group of identical specimens are presented in Table V. The average variation for all groups using the 3000-lb. concrete was 3.3 per cent while that for the 8000-lb. concrete was 4.5 per cent. The percentage variations for the specimens of 8000-lb. concrete capped with plaster of Paris were especially high although the actual variations in pounds per square inch were no greater than for the other groups of that series.

TABLE V.—AVERAGE VARIATIONS OF INDIVIDUAL TEST VALUES FROM MEAN OF EACH GROUP, IN PER CENT.

Type of Cap	Series I, 3000-lb. Concrete				Series II, 8000-lb. Concrete			
	Plane Normal Ends	Plane Beveled Ends	Convex Ends	Concave Ends	Plane Normal Ends	Plane Beveled Ends	Convex Ends	Concave Ends
Hydrostone	2.6	3.5	2.0	2.5	4.4	3.2	3.2	3.2
Castite	4.2	5.2	4.7	3.6	2.6	3.0	2.8	1.2
Oiled shot	3.0	5.1	3.7	4.8	2.8	3.5	3.0	5.1
Dry shot	4.0	3.3	3.1	3.7	2.0	4.1	4.1	5.0
Plaster of Paris at ½ hr.	1.1	3.9	2.8	3.0	5.2	6.0	4.1	9.8
Plaster of Paris at 1 hr.	2.8	3.7	2.6	3.2	3.5	6.6	5.0	13.4

SUMMARY OF RESULTS

The results of this program of tests show that both the type of capping material and the end conditions of the concrete cylinders before capping have an important effect upon the observed compressive strength of the concrete. The results may be summarized as follows:

1. Regardless of end conditions of concrete cylinders before capping, a higher strength and a greater degree of uni-

formity of strength for the same quality of concrete are obtained when using Hydrostone or Castite caps than when using the other materials tested. In general, Hydrostone is slightly superior to Castite in these respects.

2. Shot caps do not develop the full strength of the concrete. They give especially low strengths when testing specimens with concave ends.

3. In general, there is little difference between the results for oiled shot and dry shot. Of these two, dry shot is more convenient to use.

4. Plaster-of-Paris caps on cylinders of normal strength concretes having plane ends normal to the axis of the cylinder before capping develop strengths comparable to those obtained by the use of stronger capping materials. They give very low strengths for other end conditions, especially on cylinders of high-strength concrete. Even when specimens of high-strength concrete have plane normal ends, the observed strengths are relatively low.

5. In general, plaster-of-Paris caps tested at $\frac{1}{2}$ hr. produce slightly higher strengths than when tested at 1 hr., but the differences are not significant.

6. Cylinders capped with Castite may be stored under moist conditions for long periods before testing without affecting their strength adversely.

7. Considering over-all costs of labor and materials, Castite caps are cheaper than those made of any of the other materials tested.

DISCUSSION

Mr. E. N. Vidal[1] (*presented in written form*).—The subject of this paper is one of considerable importance to the laboratory technician, and Mr. Troxell is to be complimented on his excellent paper. The Bureau of Reclamation in its Denver laboratory and many of its field laboratories has used sulfur compound caps satisfactorily for many years, and the data from supporting tests should be interesting in supplementing Mr. Troxell's information.

As a preliminary activity to the prosecution of the Boulder Dam concrete research program, which started in 1931 and involved a variety of specimen sizes, it was desired to develop a capping method which would eliminate the well-recognized deficiencies of the standard neat cement cap and of the commonly used plaster-of-Paris cap. Investigation of commercial sulfur compounds and of artificial laboratory preparations led to the selection of a mixture consisting of three parts of commercial sulfur flour with one part of standard mill fire clay. Suitability, economy, and adaptability were all factors in this selection. (It has subsequently been learned that any inert siliceous material of proper fineness may be used as the filler. Waste sand, screened, has been utilized on construction projects.)

A test program was outlined to verify the adequacy of the method. 6 by 12-in. cylinders were made with $1\frac{1}{2}$-in. maximum aggregate concrete for curing in the 70 F. fog room and tested at 7, 28, and 90 days. All specimens were cast in standard cast-iron molds with machined base plates, and the tops were troweled flush with the mold. The following capping methods were included:

(*a*) For control, one group had the tops ground in a stone polishing machine on the day before test, to within 0.002 in. of a true plane as measured by a straightedge and feeler gage.

(*b*) Neat cement caps, applied in accordance with A.S.T.M. Methods of Making Compression Tests of Concrete (C 39 – 27),[2] using a normal portland cement.

(*c*) Sulfur capped, using the mixture described above, melted and poured on the top of the cylinder in a thin layer ($\frac{1}{16}$ to $\frac{1}{8}$ in.) formed by a paper strip held in place around the cylinder by a rubber band. The desired surface was obtained by pressing a warm, machined plate on the molten cap. Cylinders were capped about 30 min. before testing.

(*d*) Lumnite cement caps, same as (*b*) above except that the caps were applied 24 hr. before test and lumnite cement was used.

(*e*) Plaster caps, using a quick-setting plaster of Paris and applied 4 hr. before test.

(*f*) Plaster cement caps, same as (*e*) except that a mixture of one part plaster to two parts normal portland cement was used.

The results were as follows, each value

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[1] Engineer, U. S. Bureau of Reclamation, Denver, Colo.

[2] 1930 Book of A.S.T.M. Standards, Part II, p. 143.

representing the average for 60 test specimens:

Capping Method	Compressive Strength, psi.	Strength Comparison, per cent	Coefficient of Variation,[a] per cent
Ground	4030	100.0	6.2
Neat cement	4030	100.0	7.1
Sulfur	4070	101.0	5.7
Lumnite	4090	101.5	5.5
Plaster	3890	96.7	6.4
Plaster-cement	3930	97.5	6.1

[a] A.S.T.M. Manual on Presentation of Data, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 453 (1933).

The sulfur method was adopted for cylinders smaller than 12 in. in diameter for all except highly special tests thereafter, and larger cylinders were to be ground. Subsequently, it was decided to cap at least 6 hr. before test to insure definite hardening of the cap. It was also found that suitable caps could be formed by pouring the molten mixture on a machined plate and firmly pressing the cylinder vertically into it. The current procedure is fully described in the Bureau of Reclamation concrete manual,[3] designation 32 in the appendix.

The temperature of the molten sulfur compound has been found to be critical, as a rubbery consistency results from either over or under heating. About 340 F. has been found optimum for the mixture used. The sulfur costs $2.35 per 100 lb., and the fire clay $1.00. On an average, a cap weighs ¼ lb. for which the materials cost is one-half cent. The laboratory assistants can apply 50 to 60 caps in an hour. No attempt is made to salvage the caps from tested specimens as undesirable coarse sand is unavoidably attached, and the savings scarcely warrant the time involved. Any surplus removed during the capping operation is reused.

In 1938, the shot-cap method had been devised and used at the Grand Coulee Dam laboratory and a thorough study of this method was started to investigate its possibilities for general field laboratory use. The results of about 300 cylinders tested at Grand Coulee had indicated that the strengths of shot-capped cylinders were equal to or higher than companion cylinders which had been capped with a mixture of sulfur and sand.

The Denver tests comprised a total of 516 cylinders in seven groups, which included natural aggregates from four different deposits and crushed rock from each of two quarries. The criteria for mix proportions were that the mixture be workable and the ultimate strength at least 4000 psi. Throughout the investigation, the strength of cylinders capped by the sulfur method[3] were used as the basis for comparing all other strengths.

The studies were directed to the following:

(*a*) Comparison of the efficiency of capping media in nullifying the adverse effect of convex, sloping, or concave cylinder tops (note the difference with Troxell's treatment on both ends).

(*b*) Comparison of normally troweled cylinders for ground, sulfur, and shot caps.

(*c*) Comparison of cylinders shot-capped on top only with shot caps on both ends and a comparison with cylinders shot-capped on top only but broken upside down.

Discussion of all the test details is not necessary herein. Suffice it to record that all tests were made on 6 by 12-in. specimens and in the case of (*a*) above, the end eccentricity was ⅜ in. made by special jigs similar to those devised by Mr. Troxell.

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[3] U. S. Bureau of Reclamation Concrete Manual, Third Edition, January, 1941, Designation 32, Capping Concrete Cylinders, p. 437.

The results, in brief, are shown below:

EFFECT OF END CONDITION BEFORE CAPPING.

Capping Method	End Condition			
	Flat	Convex	Sloping	Concave
Sulfur	100	96	90	95
Shot	90	87	89	91

The benefit from sulfur caps for convex and concave ends agrees with Mr. Troxell's findings but the result with the sloping end is out of line. Shot caps occasion a reduction in strength about the same as he reported.

COMPARISON OF CAPPING METHODS.
(All ends flat before capping)

Capping Method	Compressive Strength, psi.	Strength Comparison, per cent	Coefficient of Variation, per cent
Ground	5200	102	4.0
Sulfur	5080	100	5.3
Shot	4570	90	7.6

The results agreed with the 1931 Reclamation tests, within the limits to be expected for such work, and check Troxell's comparison of sulfur and shot caps. The increased uniformity of test results for capping methods other than with shot appears significant. It may be related to the logical suggestion that the shot in spite of its small size causes point bearing with concentrations of loads during the testing operation. This was borne out by carbon imprints made in the Denver laboratory with shot-capped cylinders. The imprints also revealed that the shot which were in actual contact with the cylinder end were considerably farther apart than the diameter of the shot.

The test results for the last part of these studies were too scant to warrant definite conclusions, but it was indicated that further strength reductions were derived either from shot capping both ends or from shot capping the top and testing upside down.

Short investigations in various field laboratories validated the Denver tests and the use of shot caps has been discontinued in all major laboratories, except one. The latter has evolved a special system for shot caps which it is claimed provides results commensurate with sulfur caps. The details, however, are not available and until definitely substantiated, the shot cap should be considered subject to question.

However, shot capping has its place and can be employed in the field to advantage, for example, where occasional specimens only are involved or where facilities for handling sulfur compound are not available.

MR. W. M. MURRAY.[4]—I have been particularly interested in this report since it confirms the results of a photoelastic investigation conducted by an associate of mine, A. J. Durelli, on essentially the same problem.

In testing such specimens there are three different conditions which may prevail. First of all, one may have the desired condition in which a uniformly distributed stress is present across the ends in contact with the heads of the testing machine. In actual practice this condition is probably not very often achieved although one always tries for the best approximation to it. The second condition, which is likely the most prevalent, is that in which the friction on the head of the testing machine prevents the lateral expansion of the test specimen at its ends with the resultant development of a lateral compression in these regions. The third condition, which is most unfavorable, prevails when the capping material flows or expands laterally to a greater extent than does the test specimen. This condition produces a lateral tension and is sometimes so severe that failure takes place from tension rather than the compression that one is trying to measure. From the writer's experience this has been most noticeable in an attempt to distribute the stress on granite specimens by placing sheet lead between them and the heads of the testing machine. In this case the failure took place under about two thirds of the stress sustained without the lead and the mode of failure was by cracks parallel to the direction of the applied force indicating that a lateral tension had been developed.

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[4] Assistant Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge Mass.

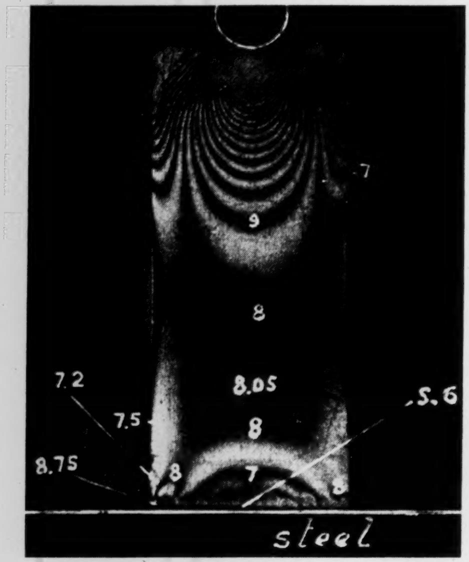

Fig. 1.—Isochromatics in a Bakelite Specimen Resting upon a Plane Steel Surface. The isochromatic concentration is about 1.1. The distribution of the stress on the boundary is far from uniform.

If one examines the results given in Figs. 2 and 3 of Mr. Troxell's paper and refers to Table I, it is seen that the best results are obtained with the two materials whose moduli of elasticity are nearest to the modulus of concrete. Also, one observes that the capping materials with strengths nearer to the strength of the concrete show more favorable results.

In general, one might draw the following conclusions from Mr. Troxell's paper:

1. That the most desirable or representative conditions can be attained with a capping material that is as strong as or stronger than the material of the test specimen.

2. That the capping material should have approximately the same modulus of elasticity and Poisson's ratio as the material under test.

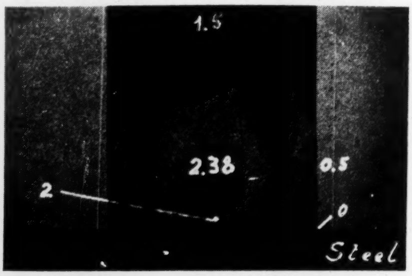

Fig. 2.—Isochromatics when the Surface of the Boundary is a Little Convex. On the corners the stresses are zero. The concentration is on the axis and its value is about 1.6.

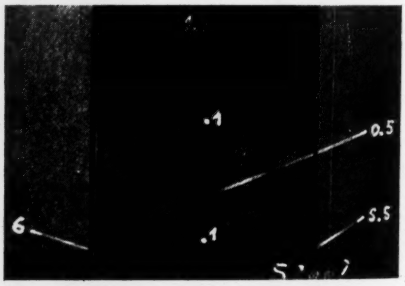

Fig. 3.—The Surface of the Model is Concave. The isochromatic concentration on the corners is about 3.8.

Mr. A. J. Durelli[5] (*by letter*).—The paper by Mr. Troxell is a very interesting contribution to the study of the end conditions in concrete test cylinders. I have been working for some time with the same problem following a different approach and using photoelastic observations.[6]

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[5] Guest in the Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

1. Rectangular specimens of bakelite with plane, convex, and concave ends bearing on smooth flat steel surfaces have been studied by the photoelastic method. The isochromatic fringe photographs are shown in the accompanying Figs. 1, 2, and 3.

The first case dealing with plane ends has been studied theoretically by Knein[7] whose results are in agreement with the photoelastic test (Fig. 4). Coker[8] made a photoelastic study of the distribution of stresses in a similar case involving a square model.

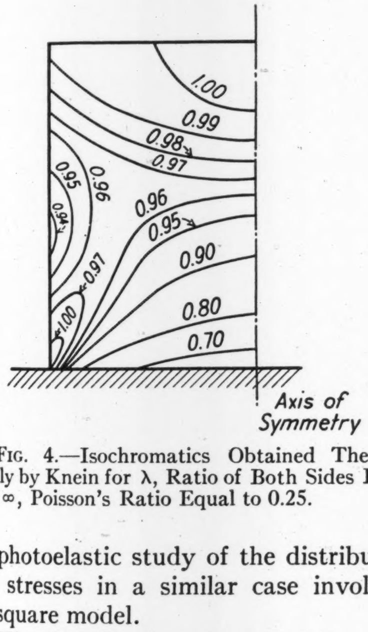

FIG. 4.—Isochromatics Obtained Theoretically by Knein for λ, Ratio of Both Sides Equal to ∞, Poisson's Ratio Equal to 0.25.

The distribution of stresses in a test cylinder of concrete is a three dimensional problem, whereas the rectangular pieces studied by Knein, Coker, and the writer give results for two dimensional distributions. However, the case of the stresses in the longitudinal central plane of the cylinder is very likely much akin to the distribution in the rectangular pieces investigated.

From these tests the following conclusions have been drawn:

(*a*) Under compression the width of the specimen is increased while the length is decreased. On the surface of contact with the steel the friction does not allow the expansion. This introduces a lateral compression on the boundary of the specimen (Fig. 5).

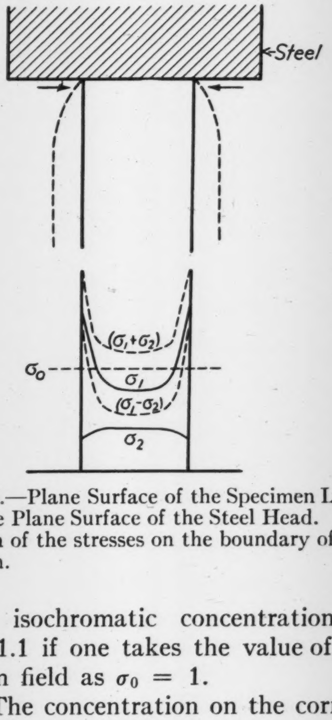

FIG. 5.—Plane Surface of the Specimen Lying upon the Plane Surface of the Steel Head. Distribution of the stresses on the boundary of the specimen.

The isochromatic concentration is about 1.1 if one takes the value of the uniform field as $\sigma_0 = 1$.

(*b*) The concentration on the corners diminishes considerably if the surface of the boundary is made a little convex. The maximum concentration is then on the axis of symmetry.

(*c*) The concentration on the corners increases if the surface of the boundary is slightly concave.

2. The same specimens were tested capped with a thin plate of lead, or sheets of rubber.

The effect of the lead or rubber is to

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[6] These results have been presented to the Thirteenth Eastern Photoelasticity Conference held at M. I. T.—"Distribution of Stresses in Partial Compression. A New Method of Obtaining Isostatics in Photoelasticity" (1941).

[7] Max Knein, "Zur Theorie des Druckversuchs," *Abhandlung*, Aerodynamischen Institut an der Technische Hochschule Aachen, Berlin, Heft 7 (1927).

[8] E. G. Coker and L. N. G. Filon, "A Treatise on Photoelasticity," p. 583, Cambridge (1931).

develop tangential stresses on the surface of contact. In this case the boundary is subjected to a tension perpendicular to the direction of the load P.

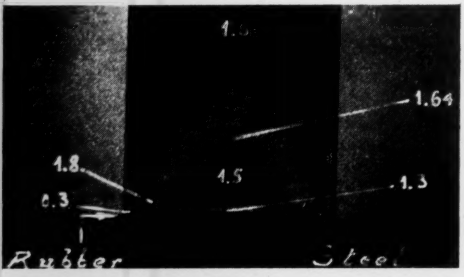

FIG. 6.—Isochromatics in a Pillar of Bakelite (2.87 by 1.32 by 0.267 in.) Transmitting the Load to a Piece of Steel (6.7 by 1.26 by 0.267 in.) Through a Sheet of Rubber, 0.027 in. Thick. The isochromatic concentration is 1.2. The value on the corner approaches zero.

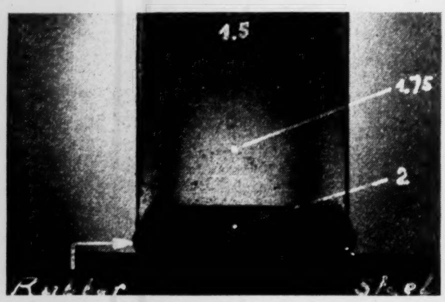

FIG. 7.—The Same Case as Before, but with Rubber 0.33 in. Thick. The isochromatic concentration is 1.33. A part of the bakelite is invisible because of the lift of the piece of rubber.

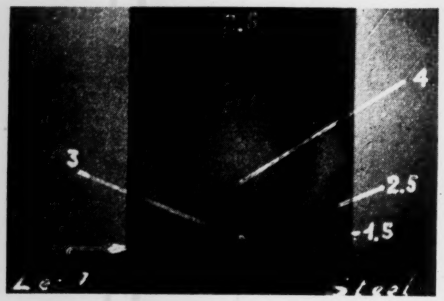

FIG. 8.—The Same Case as Before, but with Two Sheets of Lead (0.15 in.) Instead of Rubber. The isochromatic concentration is 1.14. On the corners the value of $(\sigma_1-\sigma_2)$ diminishes.

The nature of the phenomenon changes with the thickness of the rubber, or of the lead. The principal stress σ_2 on the boundary can be a compression or a slight tension. On the corners, the values of both principal stresses diminish.

The accompanying Figs. 6 and 7 show the isochromatics when the interposed material is rubber. Figure 8 represents the same bakelite model when the interposed material is lead.

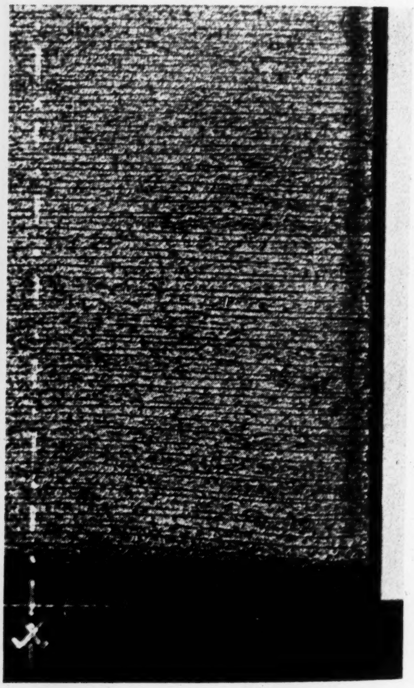

FIG. 9.—Isostatics in a Piece of Bakelite Loaded on Rubber 0.33 in. Thick. (Near the boundary the rubber destroyed a part of the lacquer.)

The isostatics are concave (looking upward) on the central part and convex near the corners.[9]

Very likely the phenomenon in three dimensions is somewhat similar. That is the reason why cubic concrete specimens, tested with rubber or grease between the concrete and the steel, have a resistance only about 25 per cent of those tested in the usual way.[10] The specimen is not broken by simple compression, but by a combination of tension and compression.

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[9] The isostatics have been obtained with the stress-coating technique which is explained in the paper presented to the Thirteenth Eastern Photoelasticity Conference.

3. The best way to obtain a uniform, or nearly uniform distribution of the stresses in a specimen, is to introduce a

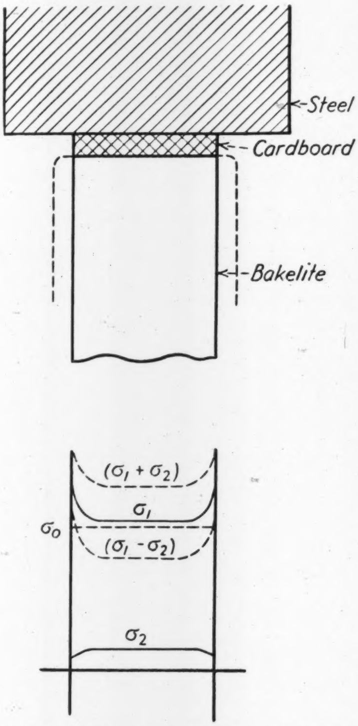

FIG. 10.—A Sheet of Cardboard Is Placed Between the Piece of Steel and the Pillar. The dotted lines represent the final shape of the pillar after deformation.

material which does not develop tangential stresses and does equalize the normal stresses. It seems that these conditions are realized very satisfactorily by using cardboard.

The effect of the cardboard is to allow a freer expansion of the boundary. The thickness of the cardboard has some influence on the distribution of the

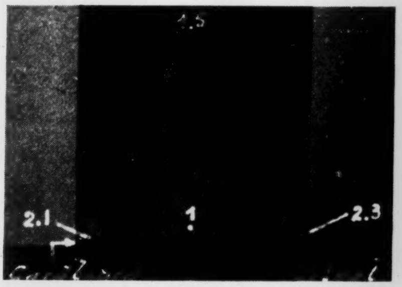

FIG. 11.—Isochromatics in the Same Bakelite Pillar (Fig. 3) Lying upon a Cardboard Sheet 0.036 in. Thick. The concentration is 1.4.

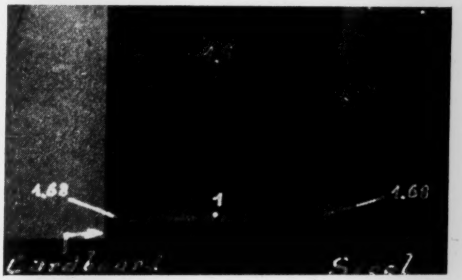

FIG. 12.—Isochromatics. If the thickness of the cardboard is increased (about 0.1 in.) the distribution is more uniform.

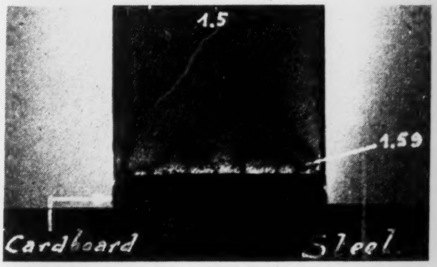

FIG. 13.—Isochromatics. When the thickness of the cardboard is about 0.25 in., the distribution of the stresses is almost uniform.

stresses. There is a thickness which produces the optimum distribution. Above and below that thickness, the concentration of stress increases. The concentration is very sharply reduced at the beginning and further increase in the thickness of the cardboard accounts for a small reduction in the concentration.

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[10] Pierre L. Brice, "Determination Experimentale et Trace de la Courbe de Resistance Intrinseque du Beton," *Science et Industrie*, Edition Construction, Janvier, 1934, p. 13.

The cardboard used was in the form of sheets about 0.036 in. thick. One of these sheets was put between the same bakelite model and the same piece of steel as examined in Fig. 3.

The maximum value has been reduced to 1.4. Three sheets of cardboard reduce the concentration to only $\frac{1.68}{1.5} = 1.12$.

The most desirable thickness seems to be about 0.25 in. Then the field is almost completely uniform. The isochromatic concentration is only 1.05. Experiments with a greater thickness of cardboard show a small increase in the concentration.

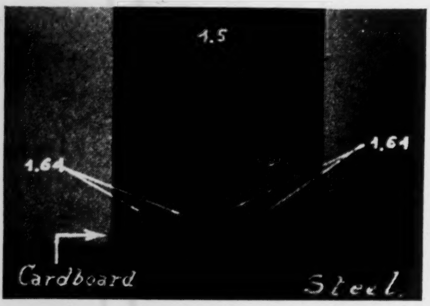

FIG. 14.—Specimen with Slight Convex Surface on the Boundary. There are six sheets of cardboard whose total thickness is about 0.2 in. The concentration in Fig. 2 has been reduced from 1.6 to 1.1.

With cardboard it is possible to obtain a uniform distribution of the stresses, even when the surface of the bakelite model is convex. The same specimen with slight convex surface, already used in the first case (Fig. 2), was tested with cardboard. The thickness of the cardboard was about 0.2 in. (six sheets). The distribution is almost uniform (Fig. 14).

4. Mr. Troxell's results for concave ends before capping are always (with but one exception) smaller than the results for convex or plane ends. That is in agreement with the photoelastic tests without capping which show a very great concentration for the concave ends. Likely the thickness of the capping was not enough to make this effect decrease or disappear.

MR. G. E. TROXELL[11] (*author's closure, by letter*).—The author is pleased to acknowledge the splendid contributions to the discussion of this paper which have been submitted by Messrs. Vidal, Murray, and Durelli.

There is just one statement made by Mr. Durelli which may require some comment. It is stated that cardboard of proper thickness serves effectively to produce essentially a uniform stress distribution on the end of the model, even though it may be concave or convex. It may be necessary to caution the reader that although this has been shown to be true when using a model of bakelite, which has a low modulus of elasticity, cardboard is definitely not satisfactory for capping the ends of a concrete cylinder which has a much higher modulus than does cardboard. As pointed out by Mr. Murray, a satisfactory capping material for a compression specimen should have a compressive strength and modulus of elasticity approximately equal to those for the test specimen.

Mr. Durelli shows that the effect of using lead or rubber to cap a bakelite model is to develop tangential stresses on the surface of contact. This is undoubtedly the same effect which is present when concrete cylinders are capped with a weak material like plaster of Paris which is subject to plastic flow. The resulting low indicated strengths for concrete capped with plaster of Paris are probably due to these tangential stresses.

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[11] Associate Professor of Civil Engineering, University of California, Berkeley, Calif.

DISCUSSION OF DYNAMIC METHODS OF TESTING CONCRETE WITH SUGGESTIONS FOR STANDARDIZATION[1]

By Leonard Obert[2] and Wilbur I. Duvall[2]

Synopsis

To assist in standardizing the dynamic methods of testing concrete, the underlying theory, apparatus, effects of moisture, and size of specimen have been studied and the findings presented herein. Errors have been found in the equations used by previous investigators in calculating Young's modulus of elasticity of concrete from the flexural frequency of vibration. A more rigorous solution of the problem has been given by Goens, the results of which are presented graphically. Experimental data are included to show the justification for using Goens equation rather than those previously used. A discussion of the apparatus used in measuring both modulus of elasticity and the damping constant is included with suggestions for improving its accuracy. A series of tests was made on concrete specimens to determine the effect of the water content and the size of specimen on modulus of elasticity and the damping constant. The results of these tests indicate that both modulus of elasticity, E, and in particular the damping constant, Q, vary with the water content of the specimen. Contrary to findings for strictly homogeneous materials, the value of E and Q for a given grade of concrete was found, for the conditions of these tests, to depend upon the size of specimen used in making the measurement, E increasing and Q decreasing as the size is decreased. Moreover, the variations in E and Q for beams of identical concrete increased with a decrease in size of specimen.

During the past few years a number of reports[3–8] have been written on the dynamic or so-called sonic method of testing building materials and in particular concrete, indicating that these methods of testing are becoming permanently established. In order that a better comparison between the results of various investigators can be made, there is need for a standardized routine of testing. This paper presents a summary of the theory, methods of testing, apparatus, and certain results of previous investigations, together with new experimental data from which conclusions are drawn as to the best methods of standardization.

The physical properties that can be most easily determined by dynamic methods are the velocity of sound, the modulus of elasticity, and the damping constant. Hornibrook[7] has shown that when a specimen is subjected to a number of freezing-and-thawing cycles modulus of elasticity shows a decrease which was correlated with physical failure of the specimen. Correspondingly, Thomson[8] has shown a similar change by measuring the damping constant. In general, the dynamic methods have been shown to be accurate and simple and have the advantage over static methods in that the method of testing does not damage or destroy the specimen.

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[1] Published by permission of the Director, U. S. Bureau of Mines.

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[3] G. Grime, "The Determination of Young's Modulus for Building Materials by a Vibration Method," *Philosophical Magazine*, Vol. 20, pp. 304–310 (1935).

[4] G. Grime and J. E. Eaton, "The Determination of Young's Modulus by Flexural Vibrations," *Philosophical Magazine*, Vol. 23, pp. 96–99 (1937).

[5] T. C. Powers, "Measuring Young's Modulus of Elasticity by Means of Sonic Vibration," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 460 (1938).

[6] L. Obert, "Sonic Method of Determining the Modulus of Elasticity of Building Materials Under Pressure," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 987 (1939).

[7] Floyd B. Hornibrook, "Application of Sonic Method to Freezing and Thawing Studies of Concrete," ASTM Bulletin, No. 101, December 1939, p. 5.

[8] W. T. Thomson, "Measuring Changes in Physical Properties of Concrete by the Dynamic Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1113 (1940).

DISCUSSION OF THE THEORY

The natural frequency of vibration of a bar is a measurable quantity which may be used to determine the modulus of elasticity. The equations relating the natural frequency of vibration to the modulus of elasticity are[9]

$$f = \frac{kV}{2\pi l^2} m^2 \quad \ldots\ldots\ldots (1)$$

and

$$V = \sqrt{\frac{E}{\rho}} \quad \ldots\ldots\ldots (2)$$

where

E = modulus of elasticity,
V = velocity of sound,
ρ = density,
l = length of specimen,
k = radius of gyration of the section about an axis perpendicular to the plane of bending ($k = \frac{t}{\sqrt{12}}$ for rectangular cross-section where t = thickness), and
m = numeric (4.73 for fundamental).

By eliminating V from Eqs. 1 and 2 and solving for E it follows that

$$E = \frac{4\pi^2 l^4 f^2}{k^2 m^4} \rho \quad \ldots\ldots\ldots (3)$$

Equation 1 was obtained by solving the differential equation for the motion of a bar vibrating in flexure in the free-free mode. Because of mathematical complexities involved, several second-order corrections were neglected in this solution, namely, the corrections for the effect of the rotary inertia, the lateral inertia, and the moment of shear. Both Mason[10] and Thomson[11] have made an approximate solution of the differential equation in which the effects of rotary and lateral inertia were included and have shown, first, that the effect of the lateral inertia increases the modulus by approximately 4 per cent, and second, that the effect of rotary inertia increases the modulus by approximately 25 per cent.

A more rigorous solution has been worked out by Timoshenko[12] and Goens[13] in which the effect of rotary inertia and moment of shear are taken into account. The results of this solution give

$$E = \frac{4\pi^2 l^4 f^2 \rho}{k^2 m^4} T \quad \ldots\ldots (4)$$

where T is a complicated correction term depending on the ratio of thickness to length and Poisson's ratio. In order to present an easy method of applying this correction as well as to eliminate the publication of complicated mathematical expressions, the value of T for different ratios of thickness to length is shown graphically in Fig. 1.

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[9] See Lord Rayleigh, "The Theory of Sound," Second Edition, Vol. 1, pp. 242–306, The MacMillan Co., London (1926).

[10] W. P. Mason, "The Motion of a Bar Vibrating in Flexure, Including the Effects of Rotary and Lateral Inertia," *Journal*, Acoustical Soc. America, Vol. 6, No. 4, April, 1935, pp. 246–249.

[11] W. T. Thomson, "The Effect of Rotary and Lateral Inertia on Flexural Vibrations of Prismatic Bars," *Journal*, Acoustical Soc. America, Vol. 11, No. 2, October, 1939, pp. 198–204.

[12] S. Timoshenko, "On the Correction for Shear of the Differential Equation for Transverse Vibrations of Prismatic Bars," *Philosophical Magazine*, Series 6, Vol. 41, pp. 744–746 (1921); also "Vibrations of Bars of Uniform Cross Section," *Philosophical Magazine*, Series 6, Vol. 43, pp. 125–131 (1922).

[13] E. Goens, "Über die Bestimmung des Elastizitätsmoduls von Stäben mit Hilfe von Biegungsschwingungen," *Annalen der Physik*, B Folge, Band 11, pp. 649–678 (1931).

The correction for rotary inertia and the correction for the rotary plus lateral inertia is given in curves 1 and 2, respectively, assuming a Poisson's ratio of 0.11. Both curves are computed from the results given by Mason[10] and Thomson.[11] Curves 3 and 4 are the corrections for rotary inertia and moment of shear assuming Poisson's ratio of 0.11 and 0.16 calculated from the results of Goens.[13] For a beam whose ratio of thickness to length is 0.25, this correction increases modulus of elasticity by 40 per cent. This correction curve is applicable to beams of rectangular cross-section only. To obtain the corresponding correction curve for a circular beam it is only necessary to multiply the scale of the abscissas by $\frac{2}{\sqrt{3}}$

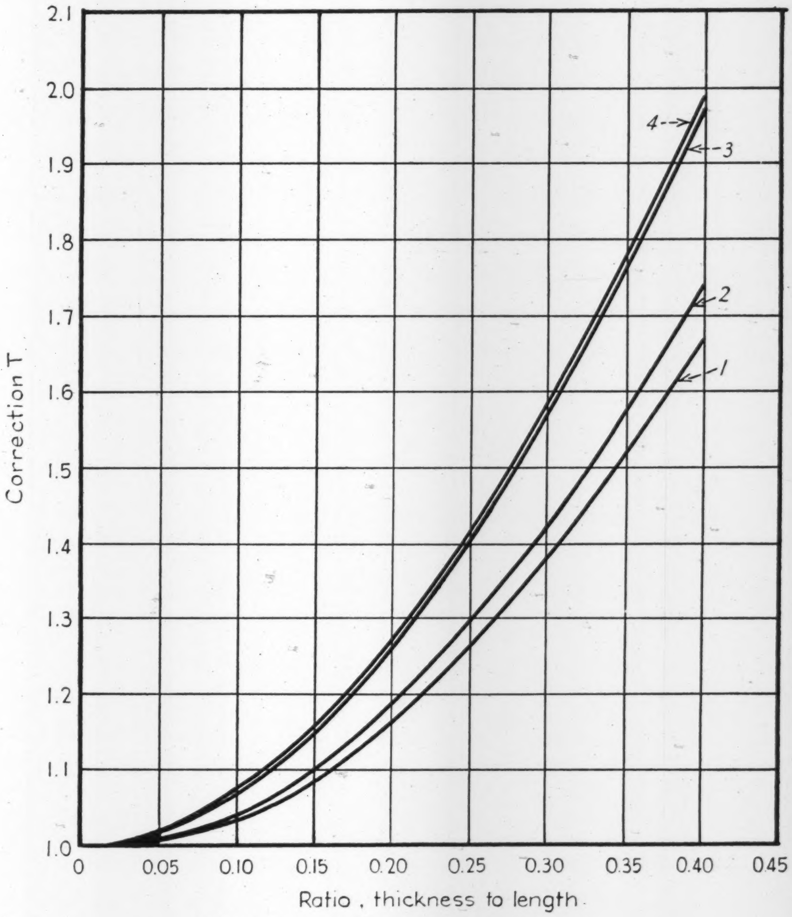

FIG. 1.—Correction Term T for Eq. 4.

Curve 1.—Correction curve for rotary inertia.
Curve 2.—Correction curve for rotary and lateral inertia—Poisson's ratio = 0.11.
Curve 3.—Correction curve for rotary inertia and shear—Poisson's ratio = 0.11.
Curve 4.—Correction curve for rotary inertia and shear—Poisson's ratio = 0.16.

(the ratio of the radius of gyration of a rectangular beam to the radius of gyration of a circular beam).

The modulus of elasticity can also be computed from the longitudinal frequency of vibration of a specimen from the following equations:[9]

$$f = \frac{Vi}{2l} \quad \ldots\ldots\ldots\ldots (5)$$

$$V = \sqrt{\frac{E}{\rho}} \quad \ldots\ldots\ldots (6)$$

where i is the integer depending upon tone of vibration ($i = 1$ for fundamental).

Eliminating V and solving for E

$$E = \frac{4l^2\rho f^2}{i^2} \quad \ldots\ldots\ldots\ldots (7)$$

In deriving Eq. 7 from the differential equation for the motion of a bar vibrating in the longitudinal mode one second-order term, correcting for the effect of lateral inertia, was neglected. If this effect is accounted for Eq. 7 becomes[9, 14]

$$E = \frac{4l^2\rho f^2}{i^2\left(l - \dfrac{i^2\pi^2\sigma^2k^2}{l^2}\right)} \quad \ldots\ldots (8)$$

where

σ = Poisson's ratio,

k = radius of gyration of section normal to axis (for rectangular cross-section $k = \sqrt{\frac{t^2 + w^2}{12}}$),

t = thickness, and

w = width.

For specimens of the size normally used in testing, vibrating in their fundamental, the term in the parentheses gives rise to a correction of the order of 1 per cent and can be neglected.

In order to show the agreement between the values of modulus of elasticity when calculated from the transverse frequency by Eq. 4 and from the longitudinal frequency by Eq. 8, several specimens of duraluminum, glass, and granite of different length-to-thickness ratios were tested. The results are given in Table I. The fifth column gives the values of the ratio of thickness to length, which range from 0.122 to 0.273. The last three columns give the corresponding values of modulus of elasticity calculated from the longitudinal frequency by Eq. 8, from the transverse frequency by Eq. 3, and from the transverse frequency by Eq. 4, respectively. The values calculated from the longitudinal frequency from transverse frequency by Eq. 4 agree within less than 1 per cent, whereas those calculated by Eq. 3 differ from the longitudinal values as much as 10 per cent.

To check the validity of Eqs. 4 and 8 for tones other than the fundamental, the transverse frequencies of vibration for the second and third tones were also measured on the duraluminum specimens as well as the first and second tones of the longitudinal frequency. Table II gives the results of these tests. The values of modulus of elasticity were computed by Eqs. 8, 3, and 4, respectively. The results show that the modulus values differ by less than 6 per cent for the different tones when computed by Eqs. 8 and 4, but decrease as much as 29 per cent when computed by Eq. 3.

The energy required to maintain a bar in sustained vibration is dissipated in two ways: first, in overcoming the internal friction of the bar, in which case the energy is converted into heat; second, that which is transferred to the external surrounding. The damping capacity, ΔW, is the energy per unit volume per cycle used in overcoming the internal friction and the specific damping capa-

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[14] C. Chree, "On Longitudinal Vibrations," *Quarterly Mathematics Journal*, Vol. 23, pp. 317–342 (1889).

city is $\frac{\Delta W}{W}$ where W is the total energy of vibration per unit volume per cycle. Analogous to electrical measurements, the Q of a specimen is given as[10]

$$Q = \frac{2\pi W}{\Delta W} \quad \ldots\ldots\ldots\ldots (9)$$

and Q will hereafter be referred to as the damping constant. Q can be determined in two ways: first, from the sharpness of the resonance curve for if f_0 is the resonant frequency and Δf the width of the resonance curve in cycles per second at $\frac{1}{\sqrt{2}}$ of the maximum amplitude then

$$Q = \frac{f_0}{\Delta f} \quad \ldots\ldots\ldots\ldots (10)$$

and second, by measuring the logarithmic decrement. The logarithmic decrement of a free vibration whose amplitude decreases exponentially is defined as

$$\delta = \log_e \frac{A_1}{A_2} \quad \ldots\ldots\ldots (11)$$

TABLE I.—COMPARISON BETWEEN THE VALUES OF MODULUS OF ELASTICITY, E, DETERMINED FROM THE LONGITUDINAL AND TRANSVERSE FREQUENCY.

Material	Dimensions, cm.			Ratio, thickness to length, t/l	Density, g. per cu. cm	Frequency, cycles per second		Modulus of Elasticity, E, dynes per sq. cm. × 10^{11}		
	Length	Width	Thickness			Longitudinal	Transverse	Longitudinal[a]	Transverse[b]	Transverse[c]
Duraluminum:										
No. 1	35.5	8.90	7.61	0.214	2.78	7120	2750	7.16	6.79	7.19
No. 2	30.70	7.64	7.00	0.228	2.78	8248	3332	7.18	6.76	7.20
No. 3	39.07	5.74	4.78	0.122	2.78	6510	1558	7.20	6.98	7.23
No. 4	27.98	8.93	7.65	0.273	2.78	9018	4200	7.29	6.81	7.31
Glass	35.00	10.18	8.50	0.242	2.56	7972	3385	8.05	7.54	8.03
Granite:										
No. 47	35.0	9.00	7.75	0.221	2.65	6010	2392	4.86	4.35	4.82
No. 48	35.0	9.00	7.75	0.221	2.65	6068	2423	4.95	4.45	4.94
No. 49	35.0	9.00	7.75	0.221	2.65	6319	2486	5.21	4.76	5.20

[a] Calculated by Eq. 8.
[b] Calculated by Eq. 3 corrected only for rotary and lateral inertia.
[c] Calculated by Eq. 4.

TABLE II.—COMPARISON BETWEEN THE VALUES OF E DETERMINED BY THE FUNDAMENTAL AND THE FIRST AND SECOND OVERTONES.

Specimen	Tone	Frequency, cycles per second		Modulus of Elasticity, E, dynes per sq. cm. × 10^{11}		
		Longitudinal	Transverse	Longitudinal[a]	Transverse[b]	Transverse[c]
Duraluminum:						
No. 1	First	7 120	2 750	7.16	6.79	7.19
	Second	13 980	6 361	7.02	5.99	7.24
	Third		10 450		5.34	7.15
No. 2	First	8 248	3 332	7.18	6.76	7.20
	Second	16 150	7 600	7.04	5.84	7.16
	Third		12 380		5.21	7.08
No. 3	First	6 510	1 558	7.20	6.98	7.23
	Second	12 960	4 008	7.21	6.63	7.33
	Third		7 082		6.25	7.19
No. 4	First	9 018	4 200	7.29	6.81	7.31
	Second		9 096		5.67	7.06
	Third		14 340		4.85	6.90

[a] Calculated by Eq. 8.
[b] Calculated by Eq. 3 corrected only for rotary and lateral inertia.
[c] Calculated by Eq. 4.

where A_1 and A_2 are amplitudes of successive vibrations. Q is related to δ by

$$Q = \frac{\pi}{\delta} \qquad \text{.........(12)}$$

Equations 10 and 12 afford two independent methods of measuring the Q of a specimen.

APPARATUS

The measurement of modulus of elasticity requires, first, a method of supporting the specimen so that it will vibrate in some prescribed mode of vibrations; second, a method of vibrating the specimen in that mode; third, a means of measuring the frequency of vibration; and fourth, a measurement of the dimensions and density of the specimen.

The method of mounting the specimen determines the mode in which it will vibrate. For the fundamental tone of the longitudinal vibration, the specimen should be mounted at the center. For the transverse fundamental tone, free-free mode, the beam should be mounted at the nodal points, which are 0.224 l from each end where l is the length of the specimen.

Both the transverse and the longitudinal vibrations have been used by previous investigators in measuring modulus of elasticity. However, for concrete, particularly when the specimen is moist and the damping losses large, it is difficult to produce longitudinal vibrations. Moreover, the longitudinal frequency is several times the transverse frequency, which also increases the difficulty in measurement.

Both free and sustained vibrations have been employed in previous investigations. The free vibrations were produced by striking the beam a light blow at the center whereas the sustained vibrations were usually produced by some type of electromagnetic device. The former method is not recommended, due to the difficulty in accurately determining the frequency of a free vibration. Further, Hornibrook[7] has pointed out that striking a specimen even a light blow might damage it if the specimen were partially deteriorated.

Several methods of measuring the frequency have been employed, the simplest of which is to match the tone of the vibrating specimen to some tone of known frequency. This method is satisfactory provided the observer possesses the ability to match tones by ear, but experience has shown that many observers will miss the true tone by as much as an octave. The other extreme is to record the vibration on a moving film oscillograph and count the waves in a given time interval. While this method is accurate it requires both excessive time and expensive apparatus. In an earlier report one of the authors[6] measured the frequency with a Syncro-clock, but this is not recommended unless extremely high accuracy is desired. The most satisfactory method is to use a calibrated audio-oscillator to determine the frequency of vibration and also to supply the power to drive the specimen. There are a number of comparatively inexpensive instruments on the market of sufficient accuracy for this purpose. The measurement of the dimensions and density of the specimen present no unusual problem except that it should be noted that the length of the specimen enters into the equation for modulus of elasticity as a fourth power and the thickness as a second power, and therefore these dimensions should be measured with care.

The damping constant of concrete has been determined by Thomson[8] by measuring the sharpness of the resonance curves. The apparatus used in making the measurement differs only from that for measuring E in that the scale on the oscillator must be increased so that it is possible to measure small changes in frequency. So far as is known no previous investigator has determined the damping constant of concrete by measuring the decrement of the free vibration.

The apparatus used in this investigation was designed to incorporate the desirable features of the apparatus previously described and at the same time eliminate any possible sources of error not previously considered. A schematic diagram of the apparatus is shown in Fig. 2. The oscillator is of the beat frequency type having a frequency range up to 17,000 cycles per second, which is sufficient to make either transverse or longitudinal frequency measurements on specimens as short as 6 in. The frequency scale can be read directly to within an accuracy of 2 per cent and a system of calibration is provided to check the frequency at 60 and 600 cycles. Whereas a frequency scale accurate to within 2 per cent is sufficient for the determination of modulus of elasticity, it is not sufficiently accurate to measure the small changes in frequency necessary to determine the shape of a resonance curve which is required for the measurement of Q. For this measurement a small variable trimming condenser was connected in parallel to the main tuning condenser. The capacity of this condenser was such that it would produce a maximum change in frequency of approximately 100 cycles per second or about 1 cycle per division of scale. In order to maintain a frequency of sufficient constancy to permit measuring a variation of 1 cycle it was necessary to enclose the oscillator in a heat-insulated box, the temperature in which was kept constant to within ±2 C.

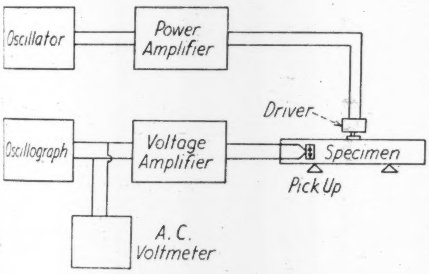

FIG. 2.—Schematic Diagram of the Apparatus.

The power amplifier was a conventional type having a power output of 8 watts, which was found sufficient for all experiments. The voltage amplifier was a conventional 3-stage, resistance-coupled type, the voltage gain of which was approximately 10,000 and was constant for frequencies from 100 to 12,000 cycles per second. The output of the voltage amplifier was measured on both a cathode-ray oscillograph and a high-impedance a–c. voltmeter. Of the two the voltmeter is more satisfactory for quantitative measurements. A moving film mechanical oscillograph was substituted for the cathode-ray whenever it was desired to photograph any results.

When damping measurements are to be made on an apparatus such as this, more consideration must be given to the method of supporting and driving the beam than for the measurement of modulus of elasticity. The energy necessary to sustain vibration in a specimen may be divided into three parts: first, the energy lost in radiation which has been shown to be negligible;[15] second, the energy required to overcome the internal friction of which the damping constant is a measure; and third, the energy lost in the interaction between the driver and the beam and the transfer of energy through the supports. Inasmuch as the damping losses cannot be separated from the losses due to the support or drive it is necessary to make the latter as small as possible.

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[15] R. L. Wegel and H. Walther, "Internal Dissipation in Solids for Small Cyclic Strains," *Physics*, Vol. 6, April, 1935, pp. 141–157.

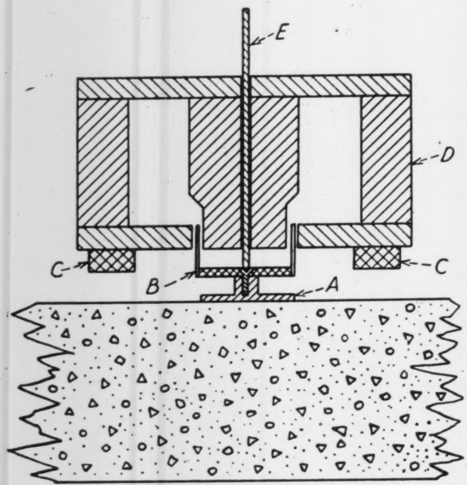

FIG. 3.—Electromagnetic Driver.

To reduce the interaction between the driver and the beam an electromagnetic system of coupling was used. In Fig. 3, *A* is a brass plate which is connected to the beam with quick-drying cement. The voice coil *B* is then fastened to *A*, and the permanent magnet *D* lowered over the voice coil until it rests on the supports *C*. The brass rod *E* was used to aid in centering the voice coil and was removed during tests. To reduce the transfer of energy through the supports, the beam was supported on piano wires accurately located at the nodal points.

Both electromagnetic and crystal pickups were used. However, due to the low impedance of an electromagnetic pickup less difficulty was encountered with stray electrical disturbances from other equipment and it was therefore used in

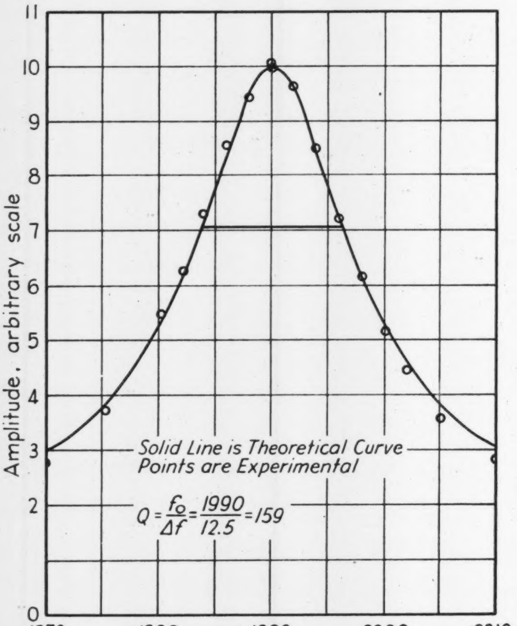

FIG. 4.—Representative Resonance Curve Showing Agreement Between Theoretical and Experimental Results.

most tests. Thomson[8] has pointed out that a crystal pickup has an electrical output proportional to the displacement, whereas for an electromagnetic pickup the output is more nearly proportional to the velocity, and therefore he recommended the crystal pickup for measuring the shape of the resonance curve. However, if Δf is small compared to f_0, which is the case for the resonance curves of concrete, it will make essentially no difference whether the amplitude or the velocity is plotted against the frequency.

To show the accuracy with which the Q measurements could be made, the experimental points for a representative resonance curve are shown in Fig. 4 together with a theoretical resonance curve having the same amplitude and Q. The estimated accuracy of the Q measurements reported herein is within ±3 per cent. Further checks were made by determining Q from the logarithmic decay of a free vibration. A specimen was first driven at its resonance frequency, then the driving force was turned off and the resulting decay photographed on the mechanical oscillograph. The amplitudes were measured and Q determined from Eqs. 11 and 12. The results of these tests are given in Table III in which the second column gives the value of Q determined from the resonance curves and the third column gives the value determined from the decay for several specimens. Of these methods the former is the more accurate and the less difficult to measure.

TABLE III.—DAMPING CONSTANT Q DETERMINED FROM THE WIDTH OF RESONANCE CURVE AND FROM THE LOGARTIHMIC DECAY OF A FREE VIBRATION.

Material	Damping Constant Q	
	Resonance	Decay
Granite No. 50	456	444
Granite No. 51	435	417
Granite No. 47	297	286
Granite No. 48	280	256
Granite No. 49	250	270
Sandstone No. 39	83	86
Sandstone No. 41	59	54
Sandstone No. 42	61	61
Duraluminum No. 1	1044	...
Glass	1390	...
Glass	(1340)[a]	...

[a] See reference No. 15.

EFFECT OF MOISTURE

In a previous paper one of the authors[6] noted that the moisture content of concrete affected the modulus of elasticity and suggested that the saturated specimen be oven dried for a given period before testing. Hornibrook[7] reported that in the first few days of air drying the saturated specimen showed a decrease in modulus of elasticity of 2 per cent whereas the overnight drying at 200 F. decreased the modulus by over 17 per cent. No study had been made on the effect of moisture content on the value of Q.

TABLE IV.—CONCRETE MIXES.

Reference Number	Cement sacks per cu. yd.	Proportions, by dry weight			Water-Cement Ratio, by weight	Flow, per cent	Slump, in.
		Cement	Sand	Gravel			
A	7	1	1.70	2.94	0.46	18.2	4.5
B	5½	1	2.55	3.59	0.61	18.7	5.7
C	4	1	4.05	4.93	0.94	21.5	3.2

In order to study the effect of the moisture content on the value of Q and at the same time make a more comprehensive study of the effect of moisture on E, the following experiments were performed: twenty-seven beams were prepared (9 each of 3 grades of concrete) substantially in accordance with the A.S.T.M. Standard Method of Test for Flexural Strength of Concrete (C 78 - 39)[16] (which specifies the method of preparing beams for flexural tests) except that the beams were rodded 50 times per layer instead of 50 times per square foot per layer. The aggregate used was Potomac River sand and gravel having a maximum size of 1 in. and the beams were cured 28 days in a constant temperature moist room before testing. The cement was a standard brand available on the local market. A description of the mixes is given in Table IV. Nine beams (3 beams of each of the 3 grades) were left in the moist room after the initial 28-day curing period, only being removed periodically for an interval of time long enough to permit testing. The second group of nine beams was oven dried at 212 F. During the initial drying period frequent tests were made and as the drying progressed the intervals between tests were increased correspondingly. The beams were removed

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[16] 1939 Book of A.S.T.M. Standards, Part II, p. 339.

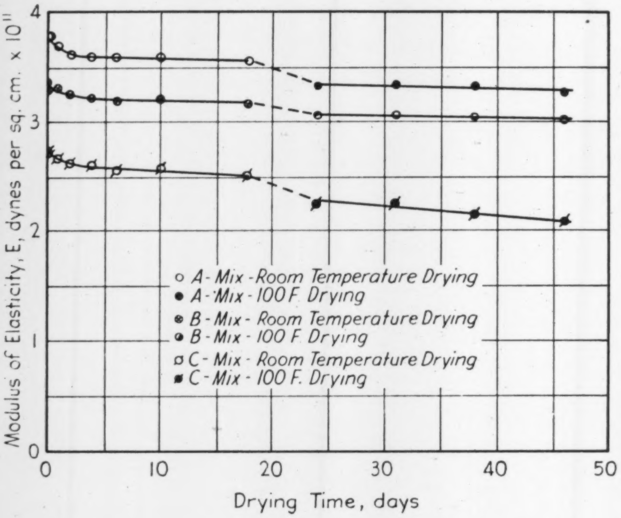

FIG. 5.—Effect of Air Drying on Modulus of Elasticity.

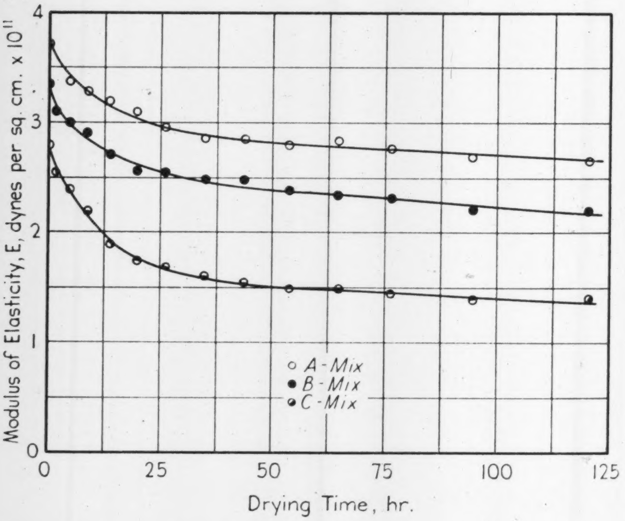

FIG. 6.—Effect of Oven Drying at 212 F. on Modulus of Elasticity.

from the oven only long enough to allow them to come to room temperature before testing. The third group of 9 beams was air dried at room temperature (65 to 75 F.) for a period of 18 days during which time tests were made periodically. At the end of 18 days the rate of drying had become very slow and to facilitate further drying, the beams were placed in a cabinet at 100 F. for 28 days, again

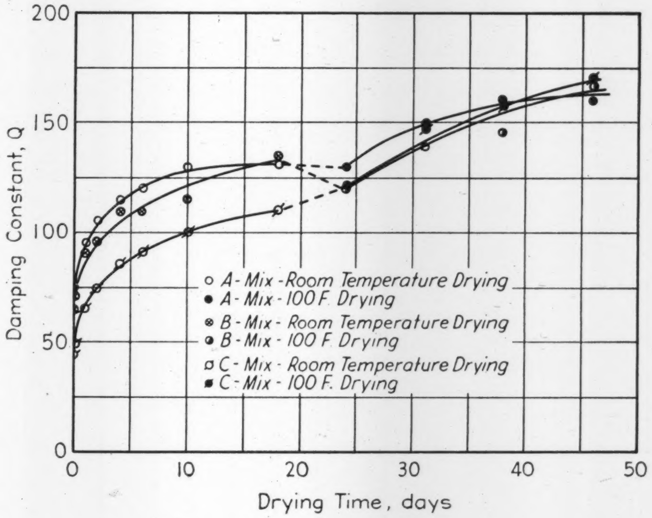

FIG. 7.—Effect of Air Drying on Q.

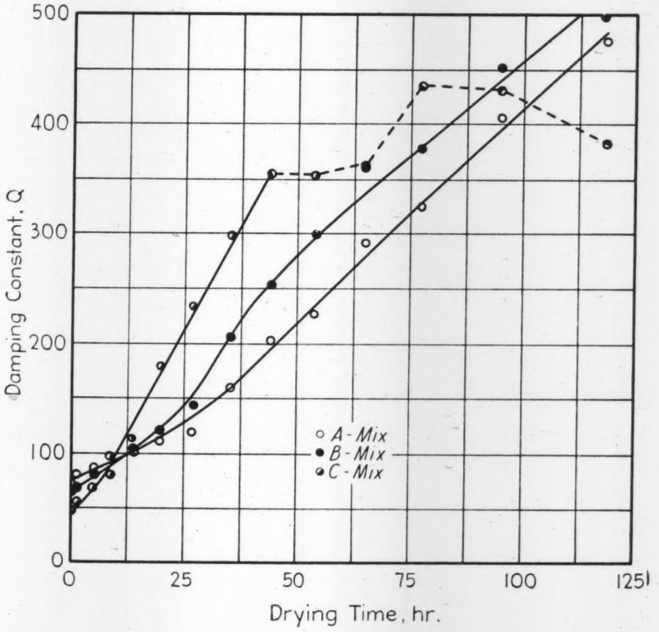

FIG. 8.—Effect of Oven Drying at 212 F. on Q.

being removed periodically only long enough to allow the beam to come to room temperature before testing. The insertion of the beam in the 100 F. cabinet produced an abrupt change in the modulus of elasticity, and for this reason both the second and third groups were soaked in water until they had regained their original weights and the drying tests repeated, except that this time the third group was air dried throughout the 37-day period.

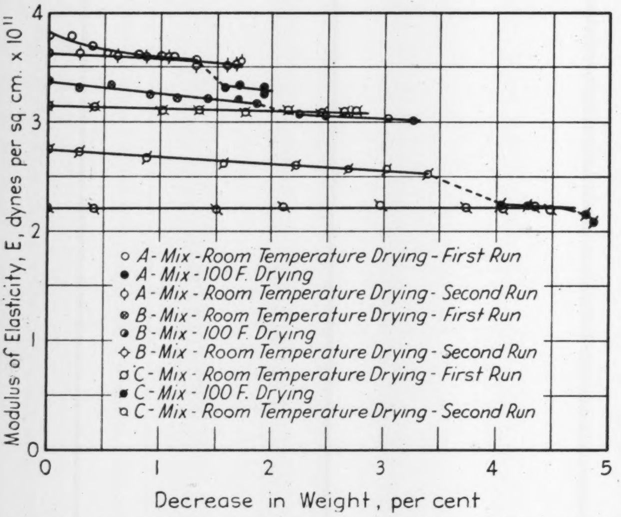

FIG. 9.—Relationship Between Modulus of Elasticity and Decrease in Weight. Air drying.

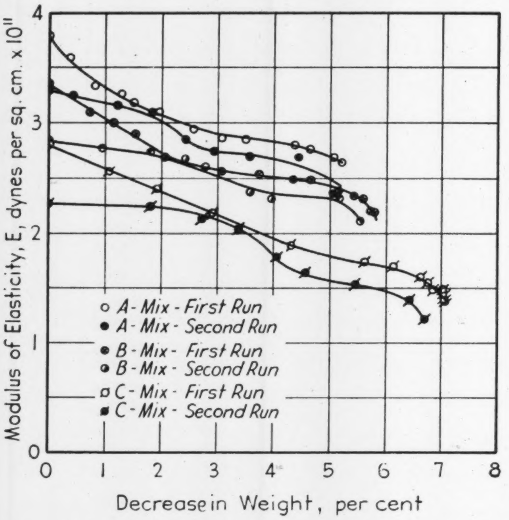

FIG. 10.—Relationship Between Modulus of Elasticity and Decrease in Weight. Oven drying at 212 F.

The results of the tests on the first group showed that E increased gradually during a 70-day period after the initial curving, the total increase being approximately 9 per cent whereas the value of Q showed practically no change. The results of these tests on groups 2 and 3 are shown in Figs. 5, 6, 7 and 8, in which E and Q are plotted against the time of drying; and in Figs. 9, 10, 11 and 12, in which E and Q are plotted against the percentage decrease in weight. In these curves each point is an average value for three beams.

Figures 5, 6, 9 and 10 show that the modulus of elasticity increases with the richness of the mix, not only for the saturated condition but for all subsequent tests throughout the period of drying. Figure 5 indicates that when the specimens are air dried there is a small initial decrease in modulus of elasticity amounting to approximately 5 per cent for all mixes, but after 3 days

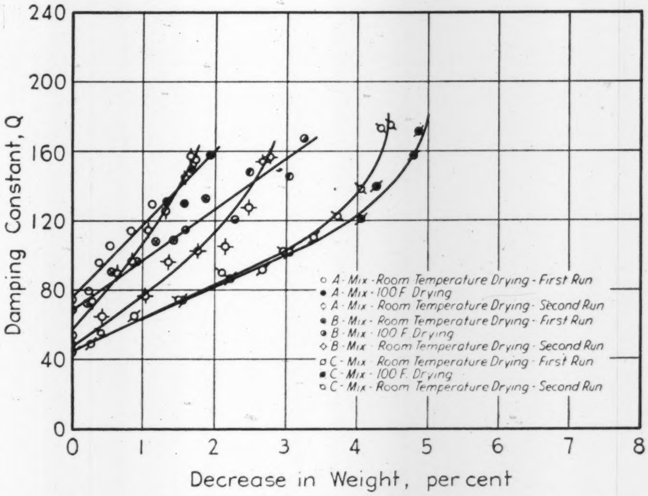

FIG. 11.—Relationship Between Damping Constant Q and Percentage Decrease in Weight. Air drying.

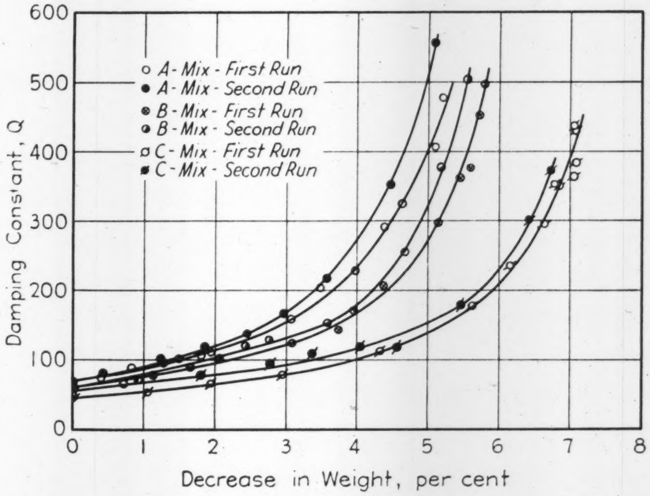

FIG. 12.—Relationship Between Damping Constant Q and Percentage Decrease in Weight. Oven drying at 212 F.

the values become nearly constant. When these specimens were first dried at 100 F. after 18 days in air the modulus decreased abruptly. However, for all subsequent drying at 100 F. the decrease was small. The largest change in modulus of elasticity during the 46-day drying period (18 days at 65 to 70 F. and 28 days at 100 F.) was for the leanest mix, amounting to a decrease of 22 per cent.

Figure 6 shows the effect on modulus of elasticity when the specimens are oven dried at 212 F. In the first 48 hr. there is a large decrease in modulus of elasticity for all grades amounting to as much as 48 per cent for the leanest mix, but after this period the rate of decrease is small.

The curve showing the change of Q with the time of air drying (Fig. 7) indicates that in the first 10 days Q increased rapidly for all grades of concrete and continued to increase throughout the remaining period, the total increase being over threefold for the leanest mix. The effect produced by drying at 100 F. is shown by the discontinuity in the curves and it is to be noted that for the subsequent 100 F. drying tests the values of Q converge for the different mixes.

Figure 8 shows the change of Q with time for oven drying at 212 F. in which the outstanding point is the rapid and nearly linear increase in Q. Also of interest is the erratic behavior of Q for the 4-sack mix after drying 44 hr. All grades show approximately sevenfold increase in Q in going from the saturated to dry condition.

In Figs. 9, 10, 11 and 12, E and Q are plotted against the percentage decrease in weight to show better the effect of the moisture content. The second drying cycle is also shown. In Fig. 9 for the initial air-drying period, E shows a small and nearly linear decrease with decrease in weight. The decrease in E when the specimens were first dried at 100 F. is still apparent, which indicates that this decrease was not due only to a large loss of water but, rather, to some effect resulting from heating the specimen. The initial point of the second-run curves shows that E will not return to its original value when resaturated. In the second run the specimens were not oven dried and the fact that these curves show no discontinuities further indicates that the discontinuities produced when the specimens were oven dried were due to temperature rather than moisture.

The effect of drying at a higher temperature compared to air drying on the modulus of elasticity of concrete is best illustrated by comparing Fig. 9 with Fig. 10. The drying curves show that E decreased more rapidly for oven drying than for air drying. It is also of interest to note that the values of E for the first and second run are nearly the same after a weight loss of 2.0 per cent.

Figure 11 shows the variation of Q with loss of weight for air drying. The effect of drying at 100 F. produces no discontinuity in these curves such as shown in Fig. 7. This indicates that Q is dependent primarily on the water content and not on any effect produced by increasing the drying temperature. This is better indicated in Fig. 12 which shows the effect of oven drying on Q. Here the first- and second-run curves are nearly superimposed, which indicates that the internal friction is dependent primarily on the water content of the specimen and not on the previous drying history. A comparison of the curve for the 4-sack mix in Fig. 8 with the corresponding curve in Fig. 12 shows that the erratic behavior shown when Q is plotted against time disappears when Q is plotted against moisture. This is due to the fact that practically no water is lost after 44 hr. of oven drying for this grade of concrete.

The free water in the different mixes was estimated[17] and it was found that the 4-sack mix had lost an amount equivalent to it after 50 hr. of oven drying, that is, about the drying time corresponding to where the value of Q first became erratic. Based on the estimate referred to, the free water had not been entirely removed from either the $5\frac{1}{2}$- or 7-sack concrete during the 120-hr. oven-drying period.

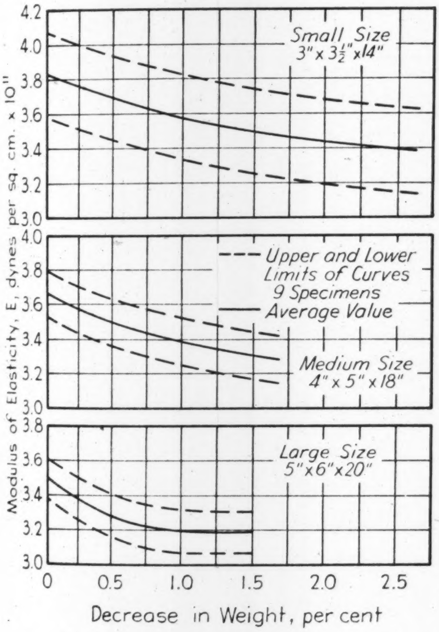

FIG. 13.—Effect of Size of Specimen on Modulus of Elasticity.

SIZE OF SPECIMEN

No mention is made in any previous report on the dynamic testing of concrete as to whether tests had been made to determine whether the size of the specimen affected in any way the accuracy of the results. It is evident that as the size of the aggregate is increased for a mold of a given cross-section the distribution of large aggregate is less random and the question arises as to what maximum size aggregate can be used in a mold of given cross-section. To study this effect, 9 beams were made of each of 3 sizes, namely, 3 by $3\frac{1}{2}$ by 14 in.; 4 by 5 by 18 in.; and 5 by 6 by 20 in. The beams were made as previously specified from the B mix, described in Table IV. The maximum size of the aggregate was again 1 in. The beams were cured for 28 days in a moist room and then removed and allowed to air dry. During the drying period measurements were made periodically of both E and Q,

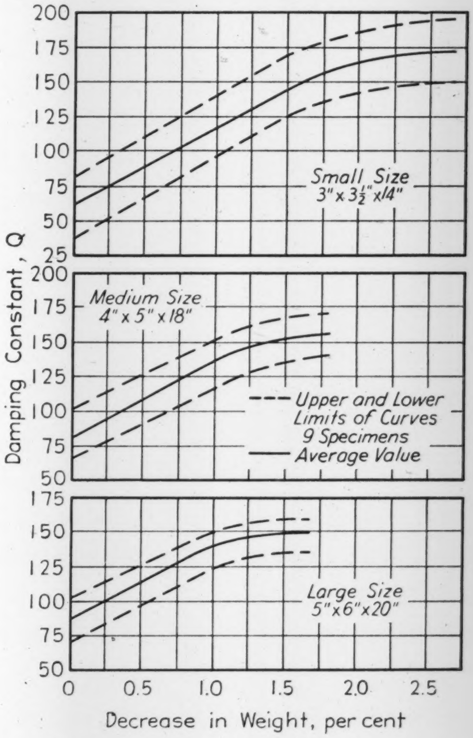

FIG. 14.—Effect of Size of Specimen on Damping Constant Q.

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[17] The free water was calculated from data given by F. R. McMillan, "Basic Principles of Concrete Making," pp. 10-12, McGraw-Hill Publishing Co., New York, N. Y. (1929).

the results of which are given in Figs. 13 and 14.

Figure 13 shows the change in E plotted against the loss in weight. The dotted curves show the upper and lower limiting curves of the 9 specimens and the solid line the average value. The 9 curves are fairly evenly distributed over the area between the limiting curves. For the 3 by $3\frac{1}{2}$ by 14-in. size, the spread between the curves for the individual specimens was 14 per cent of the average value, whereas for both the 4 by 5 by 18-in. and the 5 by 6 by 20-in. sizes the spread was 7 per cent. As the cross-section of the specimen was decreased the average value of modulus of elasticity increased, not only for the initial points but for all corresponding points on the drying curve. No explanation has been forthcoming to account for this effect.

The tests on the duraluminum beams, the results of which are given in Table I, show that for a homogeneous material the value of E is independent of the size of the specimen. On the other hand a limited number of tests on mortars, made subsequent to this investigation,[18] again showed that E increased with the size of the beam.

In Fig. 14 the spread and average value of Q is similarly presented. The curves for the large and medium-size specimens are very nearly identical. The curve for the small-size specimens shows that the spread is almost one and one-half times as large as that for the medium- and large-size-beams, and the average value is about 20 per cent lower for corresponding points on the drying curve.

CONCLUSIONS

The results presented herein were compiled with the hope that they might be useful in standardizing the dynamic method of testing concrete. It is realized that in testing concrete the diversity of experiments will necessarily limit the degree to which standardization can be achieved, but the following suggestions are presented with the hope that wherever applicable they may prove useful:

1. When the elastic behavior of concrete is studied under the influence of some deteriorating agent it is often sufficient to present the results in terms of the frequency of vibration, in which case no correction need be applied. However, if the results are compared or are to be compared with the findings of other investigators, it is desirable to present them in terms of modulus of elasticity as this is a physical property of the material.
2. If quantitative values of modulus of elasticity are determined it is necessary properly to correct the value of the modulus for the size and shape of the specimen. This can be done by using Eq. 4 and the correction curve presented in Fig. 1.
3. The agreement between the values of modulus of elasticity calculated first from the longitudinal frequency by Eq. 8 and then from the transverse frequency using Eq. 4 justifies the use of this correction.
4. The theory for the damping constant is straightforward and needs no correction.
5. The apparatus described herein was designed to measure not only the fundamental transverse and longitudinal frequency but the overtones as well, and therefore it was necessary to have a frequency range up to 15,000 cycles per second. For the usual run of concrete tests it is necessary to measure only the fundamental transverse vibration and for this the frequency range of the oscillator need only be from 300 to 3000

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[18] Made by Fred F. Bartel, Research Fellow, National Sand and Gravel Association Research Foundation, University of Maryland.

cycles per second. For convenience, it is desirable to have the oscillator dial calibrated directly in frequency, and for most tests it is sufficient that the accuracy be within ±2 per cent.

6. If Q measurements are to be made the oscillator should be provided with an increment dial for measuring small changes in frequency.

7. No precautions need be taken in supporting or driving the specimen when only E measurements are to be made. However, for Q measurements attention must be given to the drive and support to prevent energy losses through these contacts. An electromagnetically coupled driver and a piano wire support are described which reduce these losses to a minimum.

8. The E - drying time curves show that it is sufficiently accurate to measure E when the specimen is saturated. However, a better time would be after three or four days of air drying because the change of E with moisture has become small after this initial period.

9. Oven drying produces a large decrease in E during the first 48 hr., but after this initial period the change of E is small. If for any reason it is desirable to test the specimen when comparatively dry it would be satisfactory to oven dry the specimen to a given moisture content corresponding to drying time of not less than 48 hr. However it should not be overlooked that this produces an irreversible change. Oven drying, even at the low temperature of 100 F., decreases E. This decrease is not due to the loss of moisture alone, but to some unaccounted-for effect of having heated the specimen. A possible explanation of this effect is that even low-temperature drying liberates some of the combined water, the loss of which decreases the value of E.

10. The second-run tests show that when a specimen is dried and then again saturated, the value of E does not return to its former saturated value but to some lower value; hence, in tests which require the specimen to be repeatedly saturated and dried it is possible that part of the decrease in E may be due to other than the effects of the deteriorating agent.

11. The effect of the moisture content of a specimen on the value of Q is very pronounced, amounting to as much as a sevenfold increase in going from the saturated to dry condition. For this reason it is suggested that in making cyclic Q measurements the specimen be tested each cycle at some prescribed moisture content value and that the moisture content be included in the description of the test. This applies to either air or oven drying. The effect of having heated the specimen does not affect the value of Q, a fact which may suggest tests in which Q measurements would be preferable to E measurements.

12. The tests on the different sizes of the specimens show that for a 1-in. aggregate the variation in E and Q between the individual specimens is twice as large and one and one half times as large, respectively, for the 3 by $3\frac{1}{2}$ by 14-in. size as for either the 4 by 5 by 18-in. or the 5 by 6 by 20-in. size. The 5 by 6 by 20-in. beams are inconveniently heavy (weighing approximately 50 lb.) and therefore the 4 by 5 by 18-in. size is recommended for a 1-in. aggregate wherever possible.[19] Moreover, as the value of E has been shown to depend on the size of the beam, the selection of one size would unify results. The 20 sq. in. cross-section for

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[19] Rectangular rather than square cross-section is recommended so that there will be no difficulty in distinguishing between the vibration in the different planes. The approximate thickness to length ratio of 1:4 is recommended, first to keep the natural frequency between 1000 and 3000 cycles per second, and second, a ratio of 1:4 or smaller keeps the theoretical correction smaller.

a 1-in. aggregate may suggest a basis for choosing a cross-section suitable for smaller or larger aggregates.

Acknowledgments:

The concrete tests were made in conjunction with the National Sand and Gravel Association Research Foundation, University of Maryland, and all concrete specimens were prepared by them in their laboratory. The authors are deeply indebted to the association for its cooperation and in particular to Stanton Walker, Director of Engineering, whose suggestions and interest have assisted greatly in this research.

DISCUSSION

MR. GERALD PICKETT.[1]—The authors are to be commended for pointing out some defects in formulas which have been used for calculating Young's modulus of elasticity. The formula derived by Goens, which the authors have used, is no doubt the most accurate known. However, as is usually the case, the formula is still an approximation. It is based upon Timoshenko's differential equation in which no provision is made for internal friction or forced vibration. Timoshenko's derivation does not rigorously account for the interaction of shear and rotary inertia, and no provision is made for the effects of lateral inertia.

The principal effect of lateral inertia is proportional to the difference of two moments of inertia. Since this difference is zero for square cross-sections, lateral inertia may be neglected in many instances.

The effect of internal friction upon frequency may be shown experimentally by comparing:

1. Frequency of free vibration.
2. Frequency of maximum amplitude in forced vibration.
3. Frequency at which displacement and driving force are 90 deg. out of phase.

In the theoretical case of no internal friction these frequencies are identical, but as friction or damping increases they diverge. These facts should be recognized by one expecting to obtain an accurate determination of modulus of elasticity by the dynamic method.

MESSRS. LEONARD OBERT[2] AND WILBUR I. DUVALL[2] (*author's closure by letter*).—Corrections such as these suggested by Mr. Pickett would involve a prohibitive amount of computation and in view of the close agreement obtained between the theoretical and experimental results (within 1 per cent) their inclusion was not felt to be warranted.

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[1] Research Physicist, Portland Cement Assn., Chicago, Ill.

[2] Physicist, and Junior Physicist, respectively, U. S. Department of the Interior, Bureau of Mines, College Park, Md.

STATISTICAL THEORY OF THE EFFECT OF DIMENSIONS AND OF METHOD OF LOADING UPON THE MODULUS OF RUPTURE OF BEAMS

By John Tucker, Jr.[1]

Synopsis

There is an inherent difference in the strength of duplicate test specimens no matter how carefully these specimens are made or tested. Such differences are a natural characteristic of the materials and are more pronounced in some than in others. The paper shows how the variations in the strength of small elements of volume within a specimen will affect the modulus of rupture of beams of different dimensions and beams subjected to different loading. For example, the modulus of rupture of a beam will be decreased with beam length and with beam depth and will be greater in centrally loaded beams than in similar beams loaded at third points.

There has been developing within the past two decades a realization that for a given material there exists a systematic difference in the stress computed at point of failure in specimens of different size or in specimens which are subject to different methods of loading. The initial appreciation of the problem was qualitative, as illustrated by the statement of Gonnerman and Shuman (**1**)[2] that with third-point loading of concrete beams the conditions for locating by chance the weakest section of the specimen are more favorable than with cantilever and center loadings.

Griffith (**2**) in 1921 discussed the differences he found in the tensile strength of glass fibers of different diameters, and explained this variation by the presence of "flaws" on the surface of the glass. Gooding (**3**), using smaller specimens than Griffith, found the same trend. Bailey (**4**), analyzing the modulus of rupture of glass rods, established a semi-empirical relation between the strength per unit area and the size of specimens. A weakness of his method of analysis was the increasing uncertainty of the predicted strength as the length of the specimens increased.

The author (**5**) established the fundamental principles of the application of the mathematics of probabilities in correlating the compressive strength of specimens with different cross-sections and lengths, substantiating the theory with tests of concrete. Weibul (**6**) independently gave an elegant solution by generalizing and simplifying the problem, and developed mathematical equations for specimens subject to tension, compression, bending, and torsion. He simplified the solution by means of two assumptions: (1) that the strength-frequency curve is of a special type, and (2) that failure will occur in the specimen when the stress in the weakest element of volume within the specimen is the same as the stress that would cause that element, if tested independently, to fail.

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[1] Chief, Concreting Materials Section, National Bureau of Standards, Washington, D. C

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1087.

There are not sufficient data available to deduce satisfactorily the degree to which Weibul's first assumption is correct, but by its use a sufficiently accurate answer can probably be obtained. The second of Weibul's assumptions is undoubtedly correct for certain cases, such as for chains subject to tension. However, as shown by the author (5), this assumption in other cases may be incorrect and different assumptions may be valid; as for example in the case of compression, it was shown that the over-all strength of a specimen n units in cross-sectional area with proper correction for length equals the sum of the strengths of n specimens of unit area, separately tested.

This paper outlines the general and the detailed method of computing by the mathematical theory of probability the difference in modulus of rupture caused by difference in dimensions and methods of loading. It is a special purpose of this paper to develop elementary methods for these computations. Their application does not require previous knowledge of statistics or the theory of probability.

Ideal duplicate specimens to test the theory are difficult to secure and the results may be influenced by many factors. Systematic variations may be present, such as obvious differences in the concrete proportions of supposedly duplicate beams, or in the concrete within a beam. Variables may be introduced in the testing, as for example, local effects at the points of application of load, and, again, torsion may be present in different amounts in the testing of beams of different spans. The variation in the strength of the material, however, is one factor governing the modulus of rupture of beams of different dimensions and subjected to different methods of loading, and this paper attempts to evaluate some of the effects of this factor. The degree of agreement between theory and practice will be demonstrated as far as possible by citation of tests on concrete beams, but the theories presented should be applicable to other materials.

THEORY

General:

The general method of analysis consists in computing the probability of failure of the specimen under given test conditions from the strength dispersions of the elements of volume of which the specimens are composed, or of the areas corresponding to sections cut through the specimens. The elements of volume or of area are arbitrarily selected, and are chosen in such manner as to aid in the solution of the problem. The size of the elements chosen compared to that of the specimen may be very small, such as thin cross-sections of a beam, or it may be relatively large.

Preliminary to the use of mathematical probabilities in computing the effect of dimensions upon the maximum stress at failure of specimens under load, the method of failure must be assumed. Two theories of failure have been advanced by the author (5) and applied to the short column in compression. The first theory involves the assumption that failure of the specimen will occur when the stress in any one of the component elements is just sufficient to cause at least one element to fail. It should be noted that it is not assumed that failure will necessarily occur in the elements subjected to the greatest stress by the loading (as in the extreme fiber of a beam) but that it may occur in a weaker element subjected to a lesser stress. For convenience this will be designated as the weakest-link theory.

TABLE I.—VALUES OF THE MEAN AND OF THE STANDARD DEVIATION OF THE STRENGTHS OF CHAINS OF *n* LINKS.[a]

Although Tippett (8) presents these values to six figures for the means and four figures for the standard deviations, these have been reduced to two places beyond the decimal in this table, as a greater number is not significant in most experimental work.

Number of Links, n	Mean Strength of Chain	Standard Deviation of Strength of Chain
1........	M_1	σ_1
2........	$M_1 - 0.56\sigma_1$	$0.83\sigma_1$
5........	$M_1 - 1.16\sigma_1$	$0.67\sigma_1$
10........	$M_1 - 1.54\sigma_1$	$0.59\sigma_1$
20........	$M_1 - 1.87\sigma_1$	$0.53\sigma_1$
60........	$M_1 - 2.32\sigma_1$	$0.46\sigma_1$
100........	$M_1 - 2.51\sigma_1$	$0.43\sigma_1$
200........	$M_1 - 2.75\sigma_1$	$0.40\sigma_1$
500........	$M_1 - 3.04\sigma_1$	$0.37\sigma_1$
1 000........	$M_1 - 3.24\sigma_1$	$0.35\sigma_1$
100 000........	$M_1 - 4.38\sigma_1$	$0.27\sigma_1$

[a] M_1 is the mean strength of individual links, and σ_1 is the standard deviation of the strengths of individual links in the parent group of links from which the chains are made.

Weibul's analysis (6) is based wholly on this theory.

While the weakest-link theory is the only one tenable for the solution of some problems, another theory which will be called the strength-summation theory appears to apply to other problems and to give results more closely approximating test data. This summation theory involves the assumption that the strength of a specimen is equal to the sum of the strengths contributed by the component parts, or elements. Tests by the author (5) indicate that this theory more nearly agrees with compression test data for relatively short prisms or cylinders than does the "weakest-link" theory.

Under either theory it is assumed that the individual elements or imaginary subdivisions, of which the specimens are composed, are assumed to be uniformly heterogeneous (7), or drawn at random

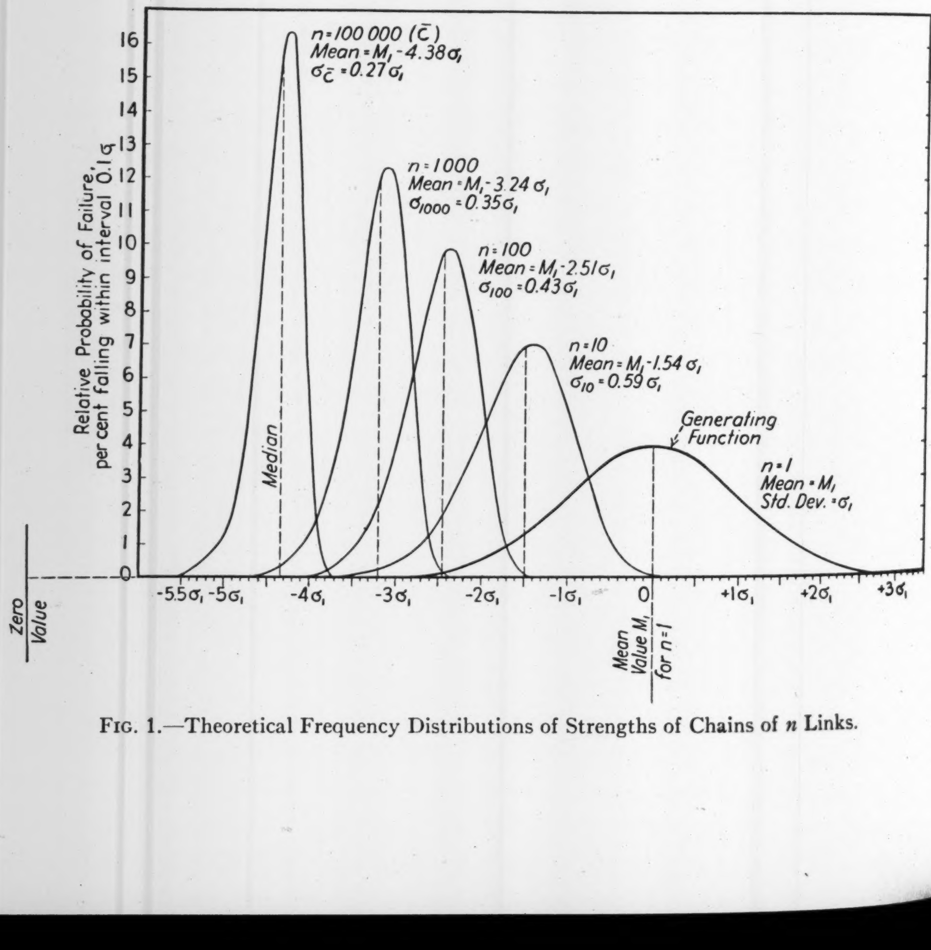

FIG. 1.—Theoretical Frequency Distributions of Strengths of Chains of *n* Links.

from the same "parent population" or parent group, in which the strengths of individual elements are distributed normally. (Solutions could also be obtained for any other distribution.)

The Weakest Link Theory:

(*a*) *Links in Series.*—As an illustration, we may select the chain, in tension, which calls to mind the well-known expression that "a chain is no stronger than its weakest link." First consider the individual links of which the chain is composed. These links in the parent group will have a mean strength M_1, and a standard deviation σ_1. If we test a group of chains of n links each, the weakest link in each chain will fail, and consequently the mean strength of chains of n links will be less than the mean strength, M_1, of the individual links. The strength-frequency distribution of chains of n links may be computed from the strength-frequency distribution of individual links in the parent group.

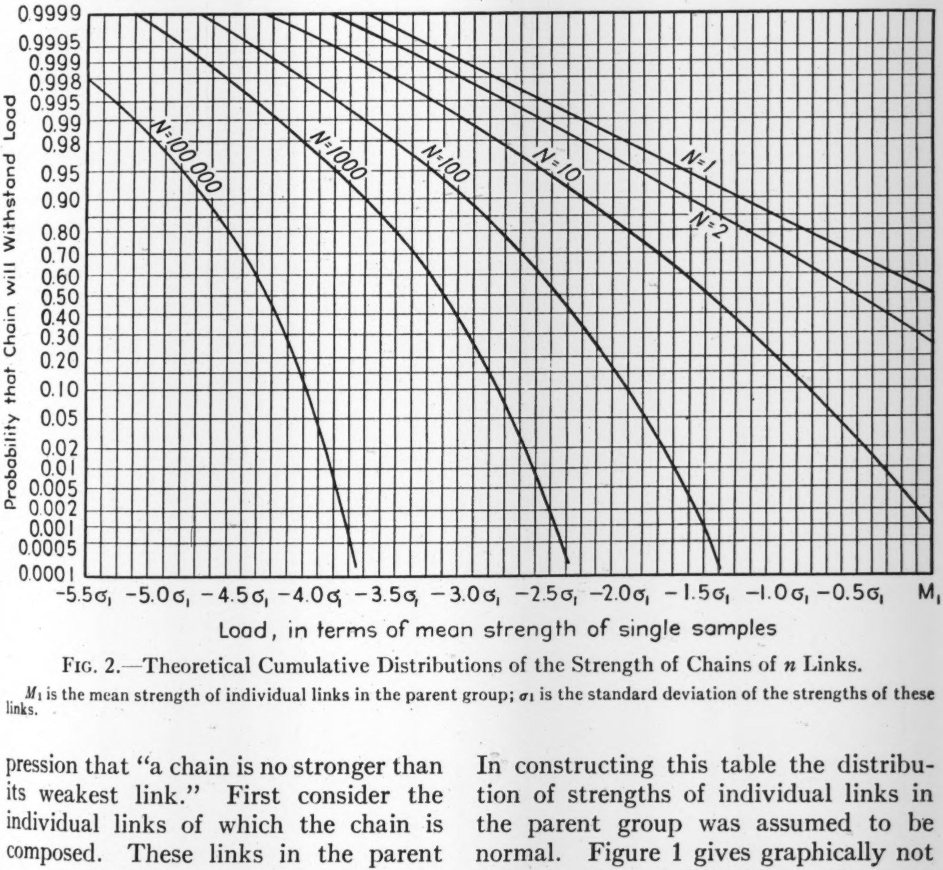

FIG. 2.—Theoretical Cumulative Distributions of the Strength of Chains of n Links.

M_1 is the mean strength of individual links in the parent group; σ_1 is the standard deviation of the strengths of these links.

Tippett (8) has given tables for the mean and the standard deviation of the least values in samples of n specimens for values of n up to 1000. The samples may be considered as chains, and the specimens as links. These means and standard deviations are given in Table I In constructing this table the distribution of strengths of individual links in the parent group was assumed to be normal. Figure 1 gives graphically not only the data of Table I but the complete frequency curves for the values of n shown. It will be observed that on the average as the number of links per chain increases, the strengths of the chains decrease, the spread (or standard deviation) of the strengths of the chains decreases, and the distribution curves for the strengths of the chains become more skew. The skewness of these curves is slight, however, and it would require many observations to detect even the skewness of the curve for $n = 100{,}000$. The mean and median values do not differ from each other materially for any of the curves. The reduction in strength of the chains with increase in the number of links is also relatively small, being only 4.35 σ_1 for $n = 100{,}000$.

The frequency curves of Fig. 1 were obtained from the cumulative frequency curves of Fig. 2. Curves similar to those given in Fig. 2 for a group of links of any number n, may be computed from the strength frequency curve for individual links in the parent group as follows:

If the probability of a single link withstanding a load M_1 is P_1, then the probability of a chain of n links withstanding the same load is

$$P_n = (P_1)^n \ldots\ldots\ldots (1)$$

To obtain the cumulative frequency curve for the chain of n links we select an adequate number of probabilities P_1 on the single-link strength probability curve and compute the corresponding P_n for each of these. To illustrate, if P_1, the probability of a single link withstanding a load M_1, is 0.5, then P_2, the probability of a chain of 2 links in series withstanding the load M_1 is $(P_1)^2 = (0.5)^2 = 0.25$. From Fig. 2 we see that P_2 has this computed value of 0.25 at the load M_1. The probability of a chain of 10 links withstanding the same load M_1 is $P_{10} = (P_1)^{10} = (0.5)^{10} = 0.000976$ which value is also indicated in Fig. 2.

From Fig. 2 we see also that the probability P_1 of a single link withstanding a load of $M_1 - 1.30\ \sigma_1$ is 0.9+. The probability of a 2-link chain withstanding this load is $P_2 = (P_1)^2 = (0.9+)^2 = 0.81+$. The probability of a 10-link chain withstanding the same load is $P_{10} = (P_1)^{10} = (0.9+)^{10} = 0.35+$.

The more the cumulative frequency curves of Fig. 2 depart from a straight line the greater the skew of the frequency curves of Fig. 1.

(*b*) *Links in Parallel.*—Let us now consider the links arranged in parallel. According to the weakest link theory the weakest link will fail when the load on that link is equal to its strength when tested as an individual link. Without further increase in the total load this failure will immediately increase the load in the remaining links by the factor $\dfrac{n}{n-1}$. The next weakest link may not fail under the resulting load; upon further increase in the over-all load a limit will be reached at which the next weakest link will fail, and so on, until the load becomes sufficient to cause all the remaining links to fail. As stated, the assembly would not *necessarily* fail at the loading which produces a load on each link equal to the strength of the weakest link. It would fail at this loading, however, if the increase in load on the remaining links resulting from failure of the first link were sufficient to cause the next weakest link to fail and this in turn the next, and so on until all the links had failed. Weibul considers this mechanism of failure, but concludes that the difference in strength from that indicated by the weakest-link-in-series theory is negligible.

The Strength-Summation Theory:

By the strength-summation theory, the strength of a parallel-link assembly of the type which has just been discussed would equal the sum of the strengths of the individual links. This theory more nearly accords with the results of compression tests on concrete cylinders of various diameters than does either form of the weakest-link theory. In this summation theory, the strength dispersion of the assemblies would increase with the number of links per assembly. If the standard deviation of the strengths of the individual links in the parent group be again designated as σ_1 then the standard deviation of the strengths of assemblies of n links would be, by the usual theory,

$$\sigma_1 \sqrt{n} \ldots\ldots\ldots\ldots (2)$$

On the other hand, the standard deviation of the mean strength of the n links would be

$$\frac{\sigma_1}{\sqrt{n}} \ldots\ldots\ldots\ldots (3)$$

ANALYSIS OF BEAMS DIFFERING IN WIDTH

Theory:

Let us assume that, according to the summation theory of failure discussed above, the strength (the bending moment required to cause failure) of a beam n units in width is the sum of the strengths of n beams of unit width. Then the mean strength of beams n units in width is nB_1 where B_1 is the mean strength of beams of unit width. If the standard deviation of the strengths of beams of unit width is Σ_1 then the standard deviation of the strengths of beams n units in width will be $\sqrt{n}\Sigma_1$.

By the theory of the strength of materials, the modulus of rupture s of rectangular beams is

$$s = \frac{B_n K}{nd^2} \ldots\ldots\ldots\ldots (4)$$

where K = a constant,
B_n = the bending moment required to cause failure of beams n units in width, and
d = the depth.

If, as according to the summation theory $B_n = nB_1$, then s becomes $\frac{B_1K}{d^2}$ and therefore for a given depth the modulus of rupture is independent of width, and is proportional to B_1.

Consequently, if σ_1 is the standard deviation of the moduli of rupture of beams of unit width, then, by Eq. 3, the standard deviation of the moduli of rupture of beams n units in width will be $\frac{\sigma_1}{\sqrt{n}}$.

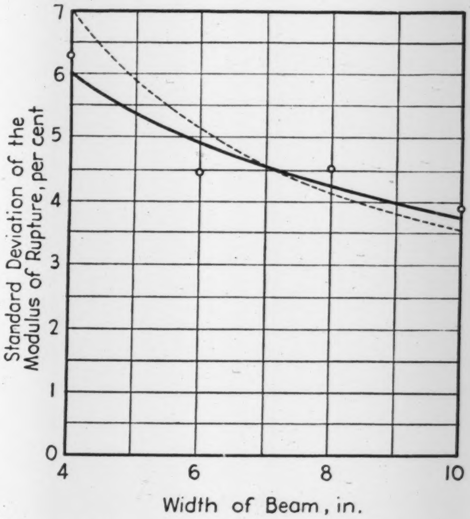

FIG. 3.—Relation of the Standard Deviation of the Modulus of Rupture to Width of Beam.

Test Data:

Reagel and Willis (9) present test data on a large group of concrete beams. The beams were of four lengths. For each length there were beams 4, 6, 8, and 10 in. in width, and for each width there were beams 4, 6, 8, and 10 in. in depth. Three beams of each cross-section and of each length were tested in each of four laboratories, a total of 768 beams of 64 sizes.

Reagel and Willis found average moduli of rupture of 801, 813, 816, and 817 psi. for beams of widths 4, 6, 8, and 10 in., respectively.

Figure 3 gives the standard deviations of the moduli of rupture of the Reagel and Willis beams of four widths, from their Table 4. Each point represents the 48 beams of square cross-section but containing four groups of four lengths. The solid curve in Fig. 3 was computed using the summation theory for the effect of width only, the effect of depth apparently having been nullified by the test method. The curve was computed from a dispersion of $\sigma_1 = 6$ per cent for a beam 4 in. square. The dotted curve was computed according to the weakest-link theory for the effect of both width and depth. The data on the dispersions do not indicate whether the weakest-link or the summation theory is superior, but clearly confirm in general the principle of the statistical theory.

The moduli of rupture of the three widest beams were slightly greater than those of the narrowest, but the difference is not significant and the tests indicate that the modulus of rupture is practically independent of width, in accordance with the summation theory. The 10-in. wide beam is 2.5 times the width of the 4-in. or unit width beam. Interpolation in Table I for a value of 2.5 gives a reduction in strength of 0.73 σ_1 by the weakest-link theory. The modulus of rupture of the 4-in. beam is 801 psi., its standard deviation approximately 6 per cent. The theoretical reduction in modulus is therefore $801 \times 0.06 \times 0.73 = 35$ psi. Thus it is clearly seen that the weakest-link theory does not apply in this case.

Gonnerman and Shuman (1) present results on three groups of concrete beams, tested as cantilevers, by center, and by third-point loading, the widths varying from 3 to 12 in. for each of the three methods of loading. The results were erratic in that the strengths at any one width, for any type of loading, varied appreciably from the average strength curves for all widths. There is, however, no indication that the width affected the strength. No data were presented on the strength dispersion for the beams of the several widths, so that the relative values of the standard deviations of the modulus of rupture at the different widths could not be calculated.

ANALYSIS OF BEAMS DIFFERING IN LENGTH

Theory:

Consider a beam of uniform cross-section, subject to uniform bending moment throughout its length. If we arbitrarily consider the beam of length l to consist of n sections of equal lengths l/n, which we will term Δ, the probability of failure within any one of these sections is the same as the probability of failure in any other section.

When failure occurs in one of the lengths, the whole beam fails, and the weakest-link theory of failure is applicable. If P_1 is the probability of any one length withstanding a given bending moment, then the probability of the whole length of the beam withstanding the same bending moment is $P_n = (P_1)^n$ (Eq. 1). From this we may compute the mean value of the moduli of rupture and the standard deviation for beams of length l in terms of the mean modulus of rupture and the standard deviation of the moduli for beams of length Δ. Hence, if the mean of the moduli of rupture and their standard deviation for beams of "unit" length Δ are M_1 and σ_1, respectively, (Table I), then since $l = n\Delta$ the mean values of these parameters for beams of length l will be those corresponding to the values for n links in Table I.

Test Data:

The beams tested by Reagel and Willis (9) were loaded at their third points. As the bending moment is uniform between these applied third-point loads, and the chance of failure occurring outside of this central portion of the beam is negligible, these beams may be considered as of length equal to the distance between the applied loads and subjected to uniform bending moment throughout their length. Reagel and Willis report for the beams an average deviation of modulus of rupture of 3.86 per cent. Using Peters' formula[3] this gives a computed standard deviation of 4.8 per cent (their Table 4). From Table I where $n = 2$ (or the beam length is doubled) the theoretical reduction in strength compared to $n = 1$ will be $0.56\,\sigma_1 = 0.56 \times 4.8 = 2.7$ per cent, or 22 psi. Reagel and Willis found that for 192 beams of 18-in. span the modulus of rupture was 817 psi., for the like number of beams of 36-in. span 806 psi., a reduction of 11 psi., half the theoretical reduction.

Gonnerman and Shuman (1) present data on cantilever, center, and third-point loaded beams varying in length up to approximately five times the shortest length. The moduli of rupture of the beams loaded at third points were unaffected by length changes but the modulus of rupture of the beams centrally loaded and loaded as cantilever showed, in accordance with our theory, a definite reduction in modulus with length. As the strength dispersion of the beams was not given, the theoretical reduction is not known.

ANALYSIS OF BEAMS CENTRALLY LOADED

Computed from Uniform Loading:

Theory:

In beams subjected to center loading the bending moment increases linearly from zero at the supports to a maximum at the center. The likelihood of failure at each point in the beam, from support to center, therefore varies, increasing from zero at the support to a maximum value at the center. The weakest-link theory is applicable for statistical analysis for as soon as the failure of the "weakest" element of length Δ occurs, the beam as a whole has failed.

The probability of the small element Δ having a modulus of rupture greater than an assigned value may be computed from test data on beams of the same cross-section and of identical material, subject to uniform loading throughout their length. From the mean modulus of rupture and its standard deviation for a beam uniformly loaded throughout its length, we may compute the similar parameters of the elementary beams of length Δ. This is the reverse of the usual procedure, and is equivalent to determining the strength and the standard deviation of the strengths of single links, from the strength and standard deviation of the strength of chains of n links (see Table I).

In the centrally loaded beam under any given loading the probability of any section Δ_i withstanding the loading to which it is subjected will be different from that of any other section Δ_j on the same side of the center. From the strength distribution of the small sections Δ we know the probability P_1 of section Δ_1 withstanding the bending moment B_1 to which it is subjected. P_2 will be the probability of the section Δ_2 withstanding the equivalent bending moment B_2 to which it is subjected, etc. The probability of one half the centrally loaded beam withstanding the loading is the product:

$$P_1 \cdot P_2 \cdot P_3 \cdots P_n = P_m \cdots\cdots (5)$$

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[3] See Johnson's "Theory of Errors," Marks Mechanical Engineering Handbook, McGraw-Hill Book Co., Inc., New York, N. Y. (1930).

The centrally loaded beam is of length $2n\Delta$. The probability of the total beam withstanding the loading is therefore, by Eq. 1, $(P_m)^2 = P_{2m}$.

We now know the probability P_{2m} of the beam withstanding the loading to produce the maximum bending moment B_n (at the center of the beam) and we can compute by Eq. 4 the modulus of rupture s_n of the beam which fails at this bending moment B_n. Hence, the probability P_{2m} of the beam having a modulus of rupture greater than s_n is now known, and we can compute the probabilities for as many values of s_n as we desire, and obtain a cumulative frequency curve.

Thus summarizing, from the strength-frequency characteristics of the uniformly loaded beam we obtain the strength-frequency characteristics of the very short beam length Δ; from these we may calculate the probability of each of the small sections Δ_i of the centrally loaded beam withstanding an assigned loading, and from these probabilities we can compute the strength-frequency characteristics of the centrally loaded beam.

Test Data:

We have test data by Kellermann (10) for beams loaded at third points. If these beams are designated as length $3L$ they are equivalent in their strength properties to beams of length L subject to uniform bending throughout. Let us now set $\Delta = \dfrac{L}{100}$ that is, small compared to L. Then from Table I we may compute the strength characteristics of the beam of length $3L$ from the characteristics of the beam of length L (the mean modulus of rupture and its standard deviation) which we know experimentally. In Table I the mean strengths and standard deviations of chains of n links are given in terms of the mean and standard deviation for individual links.

Thus from Table I we have, by analogy:

M_1 = mean modulus of rupture of beams of (unit) length Δ,

σ_1 = standard deviation of the modulus of rupture of beams of (unit) length Δ,

M_{100} = mean modulus of rupture of beams of length $L = 100\Delta$; $M_{100} = M_1 - 2.51\ \sigma_1$, and

σ_{100} = standard deviation of the modulus of rupture of beams of length $L = 100\Delta$; $\sigma_{100} = 0.43\ \sigma_1$.

From Kellermann (10) let us arbitrarily set $\sigma_{100} = 6.3$ per cent, the average value of the standard deviation of the modulus of rupture of all beams tested at third points. From Table I $\sigma_{100} = 0.43\ \sigma_1$. Therefore $\sigma_1 = \dfrac{6.3}{0.43} = 14.6$ per cent.

Consequently $M_1 = M_{100} + (2.51 \times 14.6 \text{ per cent}) = M_{100} + 36.6 \text{ per cent} = 1.366\ M_{100}$.

We have now computed the mean modulus of rupture of the beam of elementary length Δ and its standard deviation. On the assumption that the distribution of strengths of the elementary beams is normal, we can find, from the normal probability tables, the probability of the elementary beam having a modulus of rupture greater than an assigned value. As explained previously, the probability of the beam withstanding a given extreme fiber stress is the product of the probabilities of all of the small sections withstanding the extreme fiber stresses to which the beam loading subjects them. It was noted that the centrally loaded beams are of length $3L$, each half being of length $\dfrac{3L}{2}$. These beams therefore contain 300 elements of length Δ or 150 elements in one half of the beam. By using the 30 sections nearest the center, and neglecting the remainder, we get a value for probability differing by less than 0.2 per cent from that obtained by including the entire 150 sections in the computation.

rupture of beams subjected to uniform bending. This distribution is given in Fig. 4. The standard deviation of the modulus of rupture of the centrally loaded beam was computed to be 6 per cent. Kellermann found a value of 6.7 per cent experimentally.

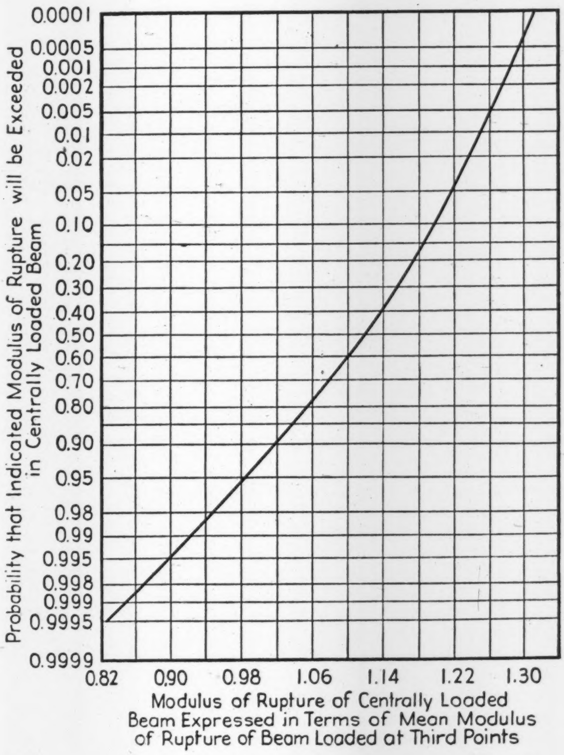

FIG. 4.—Cumulative Distribution of Modulus of Rupture of Centrally Loaded Beams.

Using data by Kellermann (10) on beams loaded at third points, the computation indicated an increase in modulus of rupture of the centrally loaded beam of 11.7 per cent above the modulus of rupture of similar beams loaded at third points. Kellermann's tests showed an increase of 13.5 per cent.

Probabilities were calculated at a sufficient number of loadings to give the frequency distribution of the moduli of rupture of centrally loaded beams expressed in terms of the mean modulus of

A normal distribution when plotted on the probability paper coordinate system used in Fig. 4, gives a straight line, and the distance along the abscissa between the point on the curve corresponding to 50 per cent probability and that corresponding to either 16.3 or 83.7 per cent probability represents by definition the standard deviation. The distribution shown in Fig. 4 is skew, and the curve is not straight. In this case a tangent drawn to the curve at the point corresponding to 50 per cent probability may be used instead of the curve itself to estimate the standard deviation with approximately correct results.

Gonnerman and Shuman (1) found a definitely larger modulus of rupture in the centrally loaded beam than they found in the beams loaded at third points. As there were no data presented on the dispersion of the modulus of rupture of the beams, the theoretical difference in modulus in the two types of beams cannot be computed.

Bailey (4) reports tests showing the modulus of rupture of centrally loaded glass rods to be 45 per cent greater than the modulus of similar rods tested at third-point loading. The strength dispersions of these specimens are not published. The dispersion of the modulus of rupture of another set of rods tested by Bailey at third points is expressed by the standard deviation of 16 per cent. The relative value of 16 per cent for standard deviation compared with 45 per cent for strength increase is not discordant with the value of 6.3 per cent standard deviation as compared with 13.7 per cent increase in modulus of rupture as reported by Kellermann.

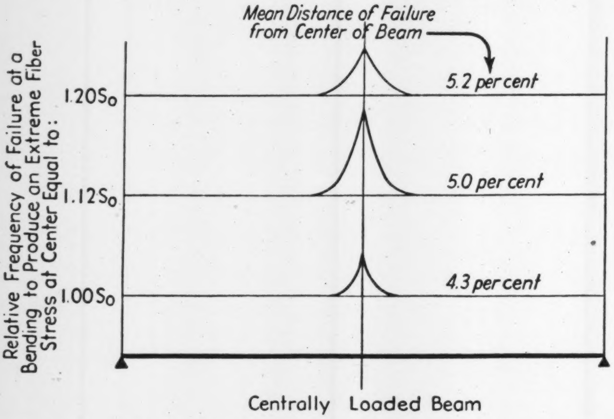
Mean Distance of Failure from Center of Beam
5.2 per cent
5.0 per cent
4.3 per cent
1.20S₀
1.12S₀
1.00S₀
Relative Frequency of Failure at a Bending to Produce an Extreme Fiber Stress at Center Equal to:
Centrally Loaded Beam

FIG. 5.—Probability of Failure Along Axis of Centrally Loaded Beam.
NOTE.—S_0 is the mean modulus of rupture of beams of same dimensions loaded at third points.

Computed from Point of Fracture:

Obviously the value of the modulus of rupture of centrally loaded beams would be less on the average if calculated from the moment at the point of fracture than if, as is customary, it is calculated at the center, where the moment is a maximum. It is now proposed to show the relation of the average modulus of rupture calculated in these two ways. This may be accomplished by the aid of computations made in determining the frequency distribution of the modulus of rupture of the centrally loaded beams. In those computations the probabilities P_i for each of the small lengths Δ_i withstanding the loading were obtained for each of a number of beam loadings expressed in terms of the mean modulus of rupture of the beam. For any given extreme fiber stress[4] at the mid-point the quantity $(1 - P_i)$ is the relative probability of failure (that is, not withstanding the loading) in the respective sections Δ_i. If these probabilities $(1 - P_i)$ be plotted along the axis of the beam in the sections Δ_1, Δ_2, etc., (where Δ_n is the section nearest the middle), the curve obtained represents the probability of failure in each of the sections.

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[4] Computed from the bending moment, upon the usual assumption of Hooke's law being valid for the material.

In Fig. 5 are plotted three curves each showing the relative probabilities of failure, at points along the axis, of the beam for a specified beam loading. The extreme fiber stresses, at the centers of the beams are, respectively, 1.20 s_o, 1.12 s_o, and 1.00 s_o, where s_o is the mean modulus of rupture of the beam loaded at its third points. These three curves are based on the probability computations used in constructing the cumulative frequency curve of Fig. 4. It is to be noted that the relative probability of failure at any location on the beam axis depends upon the load to which the beam is subjected. The relatively large area under the curve for 1.20 s_o results from the skewness of the distribution shown in Fig. 4.

The mean distance of failure from the center of the beam is given for each of the three loadings. It is not necessary to make computations for a large number of loadings; the three values given show that the mean value is approximately 5 per cent. This 5 per cent distance from the center would correspond to an equal percentage reduction in the actual modulus of rupture as compared to that computed for the center. Kellerman experimentally found an average reduction of 6.45 per cent in modulus of rupture; the reduction in modulus in the twelve sets of beams tested by him varying from 4.4 to 10.6 per cent.

Variation in Modulus of Rupture with Length of Beam:

It can be shown by similar reasoning that the variation in the strength of centrally loaded beams with variation in length is the same as for beams subjected to uniform bending throughout their length.

ANALYSIS OF BEAMS DIFFERING IN DEPTH

Theory:

(*a*) *Solution from Elementary Areas.*—Let us consider a large group of like beams subjected to uniform bending through their length. Each beam will fail under a definite bending moment, from which the modulus of rupture of each may be computed. The group furnishes the distribution of the moduli of rupture of the sections of these beams where failure occurred. Considering only the cross-sections where failure occurred, on the tension side of the neutral axis, let us imagine this area subdivided into elementary rectangles, of length equal to the width of the beams and with two sides parallel to the neutral axis, all rectangles equal in size. Let us assume that there are n such rectangles each $\frac{wd}{2n}$ in area, where d is the depth of the beam and w its width. The strain in each of these elements under the bending which just causes failure increases linearly from zero at the neutral axis to a maximum at the extreme fiber or edge of the beam. The strain will vary within the elementary areas, but we shall assume n sufficiently large so that the change in strain from one edge of the element to the other is small in value compared to the average strain in the element.

Let us now assume that Hooke's law is valid within the beam, and therefore the stresses will likewise vary linearly from zero at the neutral axis to a maximum at the extreme fiber.

The elements will all be under different stresses, increasing linearly from zero at the neutral axis to a maximum at the extreme fiber. The probability that each element of area will withstand the stress to which it is subjected will be different from that of all others, and the probability of resisting failure decreases from a maximum at the extreme fiber, to zero at the neutral axis, but not linearly. If P_1 is the probability of the element adjacent to the neutral axis withstanding the stress to which it is subjected by the applied bending moment, P_2 the probability of the second element withstanding its stress, etc., then the product of all these probabilities is equal to the probability of the section withstanding the bending moment, or, by Eq. 5.

$$P_1 \cdot P_2 \cdot P_3 \cdots P_n = P_m$$

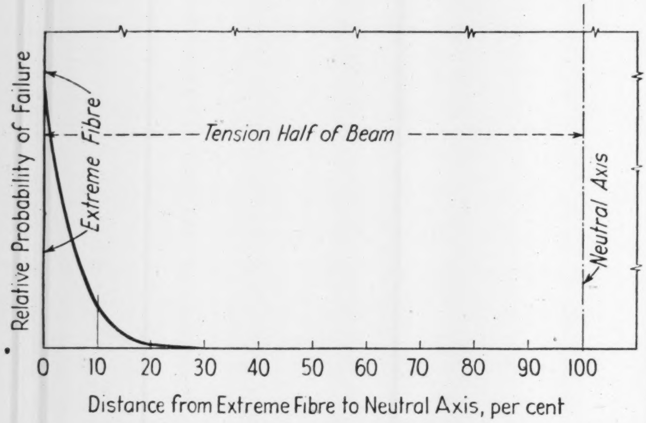

FIG. 6.—Probability of Failure at Points in Cross-Section of Beam. (Computed for 100 elements.)

In our analysis we have assumed that if failure occurs in any one element, the beam will fail, even though the element which fails is not in the extreme fiber.

We must next find the tensile strength distribution of the small rectangles, or elements of area. This distribution cannot be directly determined, but must be found by trial and error. The mean strength and the standard deviation of the strengths of these elements must be so chosen that they will give the correct value of the mean and standard deviation of the modulus of rupture of the beams.

By judicious selection of tentative values for these two quantities, the correct mean and standard deviation of the element should be determined within perhaps ten trials.

Having the distribution of the strengths of the elements, the strength characteristics of beams of any depth (that is, composed of any number of elements) may be computed.

Figure 6 shows the relative probability of failure with distance from the neutral axis for Reagel and Willis beams which fail at the mean value of the modulus of rupture. It is apparent that the probability of failure rapidly increases as the extreme fiber is approached; the probability of failure above the lowest tenth of the beam depth is very small.

It is probable that the stress in the beam section, instead of increasing linearly as has been just assumed, increases parabolically, in the well-known pattern of the stress-strain diagram for concrete in compression. An analysis of the beam upon this assumption will show *less* decrease in strength with depth than that prognosticated by the hypothesis of linear change in stress.

(*b*) *Solution from Beam of Unit Depth.*—The method of the last section is extremely tedious and not suitable for practical computations. However a method that is easy to apply may be developed as follows:

By Eq. 5, applied to a beam of any depth,

$$p_1 \cdot p_2 \cdot p_3 \cdot p_4 \cdots p_n = p_m \quad \ldots\ldots (6)$$

where p_i is the probability of any elementary area i in the beam section withstanding its stress when the beam is subjected to an extreme fiber stress s_o, and p_m is the probability of the beam as a whole withstanding this loading.

Applying Eq. 5 to a beam of twice the depth described above, for elementary areas of the same size as those of Eq. 6:

$$P_1 \cdot P_2 \cdot P_3 \cdot P_4 \cdots P_{2n} = P_{2m} \quad \ldots\ldots (7)$$

Select every odd term from Eq. 7 and we get

$$P_1 \cdot P_3 \cdot P_5 \cdots P_{2n-1} = P_o$$

and every even term, Eq. 7, and we get

$$P_2 \cdot P_4 \cdot P_6 \cdots P_{2n} = P_E$$

Hence we see that

$$P_o \cdot P_E = P_{2m}$$

Now to a close approximation,

$$\begin{aligned} p_1 &= P_1 \\ p_2 &= P_3 \\ p_3 &= P_5 \\ &\text{etc.} \end{aligned}$$

Hence P_o the product of the right-hand terms, equals, to a very close approximation p_m, the product of the left-hand terms. In this connection, note that the values of p_i rapidly approach unity with increase in i as the neutral axis is approached.

We also have:

$$\begin{aligned} p_1 &\geq P_2 \\ p_2 &\geq P_4 \\ p_3 &\geq P_6 \\ &\cdots\cdots \\ p_n &\geq P_{2n} \end{aligned}$$

Hence $p_m \geq P_E$. The difference between p_i and P_{2i} is not great for $i = 1$, and rapidly becomes smaller and negligible. P_E is an approximation for p_m differing in our computation of the last section by but a small percent and, as n increases and becomes large, P_E becomes in the limit equal to p_m.

Hence for n large $P_E = P_o$, and therefore $P_{2m} = p_m^2$. By extension of the forgoing argument $P_{km} = p_m^k$ and therefore the modulus of rupture of beams k units in depth follows the law of strengths of chains of k links. If the distribution of the modulus of rupture of the beams of unit depth is normal, then the values of Table I may be applied.

The use of Table I enormously simplifies the computation of the mean modulus of rupture of beams of depth n, from the mean modulus of rupture and its standard deviation for beams of unit depth.

Test Data:

Reagel has test data on beams 4 to 10 in. in depth. The reduction in modulus of rupture by increasing the depth two and one-half times (from 4 to 10 in.) was computed from the standard deviation of the modulus of rupture of the 4-in. beam, using the method of elementary areas described in part (*a*)[5] of the previous section. For the computation the elementary area was taken as one one-hundredth of the half-beam depth of the 4-in. beam. The 10-in. beam contained, therefore, 250 elementary areas in any cross-section.

The computed reduction in modulus was 3.6 per cent. The reduction in strength found by Reagel was 11.5 per cent, much greater than the computed reduction. However, the Reagel concrete beams were all tested with the upper surface, as cast, in tension. It would be expected that the greater the beam depth the greater the water gain at the surface and hence the weaker the upper fiber of the beam which was in tension in these tests. Such an effect would explain the greater-than-theoretical reduction in strength that was found. It is unfortunate that there are no satisfactory test data by which the theoretical reduction in strength may be checked.

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[5] See p. 1083.

Interpolating in Table I to compute the mean modulus of rupture of Reagel and Willis beams of 2.5 units in depth, by the unit depth method of the last section,[6] we find that the mean modulus of rupture of these beams should be approximately 3.5 per cent lower than the beams of unit depth. This is a sufficiently close approximation to the value of 3.6 per cent found in the previous computation.

Abrams (11) presents data showing a 6.5 per cent reduction in modulus of rupture with increase in depth of third-point loaded concrete beams, from 4 in. to 10 in. in depth. No data are available on the dispersion of the modulus of rupture of these beams, and therefore the theoretical reduction in the modulus with depth cannot be computed.

Gonnerman and Shuman (1) present data on concrete beams, showing decreases in modulus of rupture of 3 per cent, 1 per cent, and 6.5 per cent, because of increase in beam depth from 3 to $7\frac{1}{2}$ in. (increase in the ratio of $1:2\frac{1}{2}$) for beams loaded as cantilevers, centrally loaded, and loaded at third points, respectively. Again, data are not available on the strength dispersion and the theoretical reduction cannot be calculated.

SUMMARY AND CONCLUSIONS

The mathematical theory of probability has been used to compute from the mean value and the standard deviation of the modulus of rupture of beams tested by one method of loading, the same parameters of beams tested by another method of loading. Analogous computations were also made for beams of different dimensions. The computations are based upon two mutually exclusive theories of the mechanism of failure of the beams, only one of which applies in any particular analysis.

One of these was termed the "weakest-link" theory, and was assumed as a basis for computing theoretically the effect upon modulus of rupture of varying the lengths of beams and the effect of changing from central loading to third-point loading. Since a uniformly loaded beam will fail when the stresses acting within the beam are sufficient to cause failure at any cross-section, the modulus of rupture of the beam as a whole is that of the weakest section or "link." From the "weakest-link" theory, it follows that the mean value of the modulus of rupture and its dispersion decrease with increase in beam length. Various groups of data follow this pattern. In the only instance where adequate data were available upon which to base the computations, the theoretical magnitude of the reduction of modulus of rupture was greater than that found by experiment, but within experimental error. The computations indicated that the modulus of rupture of centrally loaded beams is theoretically greater than that of beams of equal length loaded at third points. The data confirm the theory. The computed average distance of the plane of rupture from the center of the beam was verified experimentally.

The weakest-link theory was applied also, for lack of any better hypothesis, in the problem of the effect of beam depth upon modulus of rupture. In this case, however, a cross-section of the beam was considered not as intersecting a single link, but as intersecting layers of links or fibers. It was assumed that one of the internal fibers, if relatively weak, may fail before an outer fiber, even though the stress is greater at the outside, and that the failure of one such fiber will result in failure of the beam. If failure always originated at the outer fiber, then, according to our analysis the beam strengths and strength dispersions would be far greater than they are. The computations indicated that the modulus of rupture of beams decreases with increase of beam depth. This conclusion is in agreement with experiment; but since the dispersion in the data is not reported the theoretical and the actual reduction cannot be compared.

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[6] See p. 1085.

The second theory was termed the "summation" theory, and was assumed as a basis for computing theoretically the effect of variations in the width of beams upon their strength and strength dispersion. Under this assumption, the strength of a specimen is equal to the sum of the strengths contributed by the component parts or elements. This theory was used for width variations because it gave results more in accord with available data than were computations based upon the "weakest-link" theory. On the basis of the "summation" theory, the modulus of rupture of beams would not be changed with variation in width; but the dispersion of the modulus of rupture would decrease with increase in width. Experimental results are in reasonably good agreement with the theory.

It was shown that increase in the size of specimens may cause little or definitely no reduction in the modulus of rupture (as with increase in width) or it may cause reduction in modulus of rupture (as with increase in length or depth of beam). The dispersion of the modulus of rupture of the specimens is reduced in either case. For engineering structures, large in size in comparison with test specimens, the dispersions of the modulus of rupture will be relatively small, thus making a comparatively high degree of uniformity.

Summarizing, the mean value and the standard deviation of the modulus of rupture of beams of concrete or other uniformly heterogeneous (7) materials is affected by change in beam dimensions as follows:

1. The modulus of rupture is independent of the beam width. The standard deviation of the modulus of rupture of a beam n units in width is $\frac{\sigma_1}{\sqrt{n}}$ where σ_1 is the standard deviation of the beam of unit width.

2. Increase in length, or increase in depth of beam causes decrease in the modulus of rupture and decrease in its standard deviation. These decreased values are the same as those for the least values in groups of n, as given in Table I.

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DISCUSSION

MR. GERALD PICKETT[1] (*presented in written form*).—Mr. Tucker has very clearly stated two statistical theories for the effect of dimensions upon the modulus of rupture of beams. He has questioned the application of the weakest-link theory to elements in parallel and has proposed instead the strength-summation theory. He is to be commended for this step. His analysis of beams differing in width on the basis of this summation theory is excellent. However, the writer sees no reason why the theory cannot also be used for beams of varying depth and thereby have a wider application than claimed by the author.

Unfortunately the author abandons his logical summation theory for beams of varying depth because it does not appear to fit the data and applies the weakest-link theory "for lack of any better hypothesis."

This lack of agreement is more apparent than real. The chief source of the error lies in our method of computing the modulus of rupture, and therefore the data from various sources were not in the proper form for his mathematical analysis. An important error in modulus of rupture data is explained below.

One of the assumptions made in deriving the elementary flexure formula is that the beam is subjected to pure bending. This means a constant bending moment with external forces applied only at the ends and distributed in a prescribed manner. Loads applied at the upper surface and supports at the lower surface cause deviations from the elementary theory. When the length to depth ratio is large these deviations are small but this ratio is usually not large for laboratory specimens of concrete.

The correction in the flexure formula for the effect of length to depth ratio is treated by Karman and Seewald.[2] In the case of third-point loading the stress in an outer fiber at a section between the loads becomes

$$\sigma = \frac{P}{Wd}\left[\frac{L}{d} + \beta_1 + \beta_2 + \beta_3 + \beta_4\right]$$

where the first term in the bracket is identified with the elementary formula and the β's are corrections which may be taken from the curves on page 23 of Seewald's paper. The corrections β_1 and β_2 for the two loads are given by his $\gamma_5\ (t)$ curve and the corrections β_3 and β_4 for the reactions are given by his $\gamma_1\ (t)$ curve. For such a loading the theoretical maximum tensile stress does not occur at a section midway between the loads except for a span-to-depth ratio of less than about two. For ratios greater than about two, calculations show that sections closer to the loads than the mid-section have slightly larger stresses. However, the error will be small and the work simplified if the stress at the mid-section is taken as the maximum.

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[1] Research Physicist, Portland Cement Assn., Chicago, Ill.

[2] Th. V. Karman and F. Seewald, *Abhandlungen* aus dem Aerodynamischen Inst. an der Technischen Hochschule, Aachen, Vol. 7, pp. 3-33 (1927).

Using Seewald's curves and calculating for an 18-in. span it is found that the tensile stresses of an outer fiber at the mid-section for 10- and 4-in. beam depths are approximately 11 per cent and 1 per cent greater, respectively, than that given by the elementary beam formula. Such theoretical values have been verified by photoelastic methods such as for recent work of Jakkula[3] of the Texas Highway Dept.

If the data used by the author had been corrected for the effect of length to depth ratio, better agreement with his theory would have been obtained.

The author may have been puzzled by the data of Reagel and Willis[4] which shows practically no effect of length upon modulus of rupture if the cross-section is not changed. The weakest-link theory which applies in this case because the sections are in series would indicate a lowered modulus but in reality this is offset by the effect of the increased length to depth ratio.

MR. G. R. GAUSE[5] (*by letter*).—Mr. Tucker's paper presents a point of view which should be of much interest to all who are concerned with the testing of structural materials and in particular to those whose work is the standardization of test methods. His paper has demonstrated that, on the basis of certain assumptions, the expected unit strength of a test specimen is a function of the size of the specimen. Although some of the assumptions made may not be valid in some cases, it is not unreasonable to believe that in certain cases the assumptions are satisfactorily met. And it is just these cases which make it imperative that anyone engaged in testing the strength of materials should have in mind the basic ideas underlying Mr. Tucker's theory.

There seem to me to be three practical contributions in this paper:

First, the necessity of standardization of test methods is demonstrated once again, and this time from a consideration of factors not often taken into account. In order to ensure that different laboratories are measuring the same thing it is necessary that they use specimens of the same size and shape, and also that they use the same methods of loading. This fact has of course long been realized by many, but the explanation has often been hazy and unconvincing. Mr. Tucker's paper demonstrates one reason why standardization of test methods is necessary.

Second, the necessity of reporting some information as to the variability of test results is exemplified. The Society's Committee E-1 on Methods of Testing has published an excellent Manual on the Presentation of Data. This manual gives methods for presenting the most important information about research data in the most condensed form. If such methods are followed in technical papers, many new theories such as Mr. Tucker's can be tested without recourse to additional tests.

Third, another step has been taken in the clarification of our thinking with regard to the strength of a material. From the classical viewpoint a structural member is made up of a homogeneous material, and its ultimate strength is completely determined by one constant of the material. From the statistical viewpoint the member is made up of a number of small parts or "links" and the ultimate strength of the member is a function of at least two constants of the material, the mean strength of the "links" and the variability of the "link" strengths. It is only when the strength of each "link" is exactly the same as the strength of every other "link" that the statistical viewpoint coincides with the classical.

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[3] A. A. Jakkula, "An Investigation of Short Beams with Center and Third Point Loading," Texas Highway Dept. Report, September, 1940.

[4] F. V. Reagel and T. F. Willis, "The Effect of the Dimensions of Test Specimens on the Flexural Strength of Concrete," *Public Roads*, Vol. 12, No. 2, April, 1931, p. 37.

[5] Associate Statistician, Aberdeen Proving Ground, Aberdeen, Md.

In comparing Mr. Tucker's treatment of the statistical viewpoint with the treatment used by Weibull[6] the outstanding difference to be noted is the introduction by Mr. Tucker of the so-called "strength-summation theory." As stated, this theory appears to apply to certain problems and to give results which agree better with test data. However, certain applications of this theory will lead to inconsistencies. For example, suppose we have a number of beams of width w and length l whose mean strength is M and whose strength standard deviation is σ. We ask the question: "What is the mean unit strength of beams of width $2w$ and length $2l$?" According to Mr. Tucker's method we may proceed in two ways to compute the mean strength of the larger beams. First we may compute the mean strength and standard deviation of beams of length $2l$ and width w. This would be, according to Table I, $M_{2l,\,w} = M_1 - 0.56\sigma_1$; and $\sigma_{2l,\,w} = 0.83\sigma_1$. We would then compute the mean unit strength of two of these beams in parallel. This would be, according to the strength-summation theory, the same as that of each beam of length $2l$ and width w. That is, $M_{2l,2w} = M_1 - 0.56\sigma_1$. The other method of proceeding would be first to compute the mean strength and standard deviation of a beam of length l and width $2w$. According to the strength-summation theory this would be $M_{l,2w} = M_1$; and $\sigma_{l,\,2w} = \sigma/\sqrt{2}$ Then compute the mean strength for two of these beams in series. This would be according to Table I:

$$M_{2l,2w} = M_{l,2w} - 0.56\sigma_{l,2w}$$

$$= M_1 - \frac{0.56 \times \sigma}{\sqrt{2}}$$

Thus by the second method of computation the reduction in strength is only $1/\sqrt{2}$ times as much as it is by the first method of computation. In the same manner we find that the reduction in strength of a beam of length ml and width mw is, when computed according to the second approach, only $1/\sqrt{\ }$ times as much as when computed according to the first approach. The inconsistency lies in the fact that two different assumptions as to the mechanics of failure have tacitly been made in following the two approaches. According to Mr. Tucker's method one would follow one or the other approach depending upon what data one happened to have at hand. This would of course lead to inconsistencies. On the other hand Weibull's treatment[6] which considers nothing but the "weakest-link theory" is consistent in itself and hence is satisfactory from a mathematical or logical viewpoint, the only question being, does the "weakest-link theory" assumption always hold? It seems certain that in some cases the "strength-summation" theory holds, but it remains to be shown how the theory can be applied with consistent results to specimens which simultaneously vary in width and length.

With regard to the assumption by Tucker of a normal frequency distribution and by Weibull of a "special type" distribution, it is worth while to remember that the normal distribution is so often used not because it alone exactly represents the data but because it represents the data satisfactorily enough for the purpose at hand and because the assumption of normality simplifies subsequent mathematical computations. In the statistical treatment of the strength of materials Weibull's assumption of a "special distribution" simplifies the subsequent mathematical computations and hence enjoys in this field that property which in the field of theory of errors is enjoyed by the normal distribution assumption. If this "special distribution" is found to agree with experimental data then it is to be preferred over the normal distribution in applications of the statistical theory of strength of materials.

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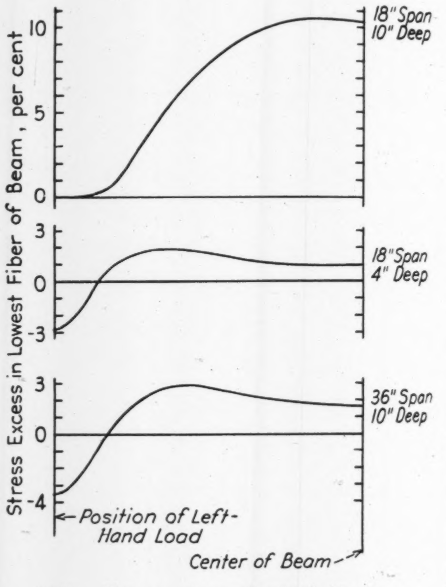
[6] W. Weibull, "A Statistical Theory of the Strength of Materials," *Proceedings*, Royal Swedish Inst. Engineering Research, No. 151, pp. 1–45 (1939).

Mr. Tucker has advanced a theory which carries important implications and his paper should elicit further investigations both theoretical and experimental.

MR. JOHN TUCKER, JR.[7] (*author's closure, by letter*).—The modulus of rupture of a beam, as computed by the mathematical theory of probability from the stress distribution predicated by the standard Euler-Bernoulli analysis is influenced by numerous factors, such as the departure of the material from true compliance with Hooke's law. Moreover, variation in the modulus of elasticity from point to point within this material would, by the theory of probability, cause variation in the location of the neutral axis, thus making the statistical analysis of beam strength still more complicated. It was, however, deemed unwise, in a paper developing the subject, to introduce any unnecessary complications, and therefore the analysis was limited to one based on the stress distribution of Euler-Bernoulli.

Seewald's little-known stress corrections, so pertinently cited by Mr. Pickett, are of a magnitude to cause considerable error in the modulus of rupture values for the shorter beams discussed in the paper. The Seewald theory is also valuable in explaining the experimental tendency of short beams loaded at third points to break at sections away from the loads. This is evident from the accompanying Fig. 1 which gives the excess stresses as computed from Seewald's curves, for the extreme lowest fiber for beams of 18 and 36 in. spans, and of 4 and of 10 in. depths.



Note.– Excess stress for 36-in. beam, 4-in. deep, is negligible

FIG. 1.—Seewald's Corrections for Stress in Lowest Fiber of Beams Loaded at Third Points.

On first thought, the correction which should be applied to values of the modulus of rupture computed by probability theory would be the maximum excess as computed from Seewald's curves; this would, it is true, be the correct procedure for the classical treatment where each unit volume of the material is assumed to have the same strength as every other unit volume. However, in every known material the strength of each unit or element of volume varies. In a beam of such a material in which the stresses vary (as, for example, the extreme fiber stresses in the centrally loaded beam which vary linearly from zero at the support to a maximum in the center) the author's paper has demonstrated that in order to obtain the mean modulus of rupture of the beam it is necessary to compute the probability of failure of the beam from the probability of failure of *every* short elementary section in the beam.

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[7] Chief, Concreting Materials Section, National Bureau of Standards, Washington, D. C.

In applying Seewald's corrections, we again cut the beam into elementary lengths, Δ, as in the centrally loaded beam, p. 1079. From the probability of each elementary length Δ withstanding the given loading we compute the probability of the beam withstanding the given loading. We should take cognizance not only of the *extreme* fiber stress in each section, but of the stress distribution across the beam sections, which, according to Seewald's analysis, will also vary from that distribution computed from the Euler-Bernoulli hypothesis. However, in computing the values given later, this latter stress distribution has been neglected and it has been assumed that the distribution is linear for each section, with maximum fiber stress in that section as given by Seewald.

The computed decrease in mean modulus of rupture because of the Seewald effect is as follows:

Beam Length, in.	Depth, in.	Decrease in Modulus of Rupture, per cent
18	4	1.0
18	10	7.5
36	4	0
36	10	1.5

The decrease in modulus of rupture caused by increasing the depth of beam from 4 in. to 10 in. is 6.5 per cent in an 18-in. span, 1.5 per cent in the 36-in. span. As the Reagel and Willis tests included an equal number of beams of 18-in., 24-in., 30-in., and 36-in. span, the mean decrease in modulus of rupture caused by increasing the depth from 4 to 10 in. is approximately 4 per cent. This quantity, added to the 3.5 per cent, found previously, and based on the Euler-Bernoulli stress distribution gives a 7.5 per cent decrease in strength, much closer to the experimental value of 11.5 per cent. It must be noted that the scale of the curves given by Seewald is so small that values read from them are only approximations, and the computations based upon them must be considered also as approximations.

Mr. Gause presents a most important problem in citing the inconsistency which arises when the modulus of rupture of beams of increased depth, length, and width is computed by statistical theory in two ways from the modulus of rupture of beam of the original size. Until this matter is cleared, the statistical theory of the strength of beams as propounded will not rest on a satisfactory foundation. As Mr. Gause points out, Weibull's theory gives mathematically consistent results, but on the other hand the summation theory appears to be experimentally indicated. No wholly satisfactory solution of the problem may be obtained until sufficient experimental evidence is obtained to clarify the situation.

One factor already mentioned should have an appreciable effect on the calculation of the modulus of rupture of various size beams. That factor is the indeterminate location of the neutral axis of the beam because of the variation in the *modulus of elasticity* throughout the beam. Heretofore we have only considered the variation in the *strength* of the material. It may be that if the theory is broadened to include the effect of variation in modulus of elasticity, the discrepancy cited by Gause would disappear.

The author is greatly pleased to have elicited the excellent criticisms pertaining to both statistics and mathematical stress analysis. These criticisms have greatly added to the paper.

PERFORMANCE SPECIFICATIONS FOR GREASES[1]

By Robert C. Adams[2] and Harrison E. Patten[2]

Synopsis

Special testing equipment and methods for its use are described in connection with the performance specifications which are being developed by the Navy. Plasticity, leakage, bleeding, and resistance to oxidation are measured under accelerated or simulated service conditions and the specification requirements are based upon these results instead of upon composition. Performance testing permits the identification and purchase of superior greases with reduction in costs of lubrication despite some increase in unit cost.

The Navy has known for several years that the specifications under which the purchases of lubricating greases are made are faulty and has been searching for an improved method of specification. Increasing speeds, bearing loads, tooth pressures and operating temperatures have increased the demands on lubricants, in many cases beyond the point where simple composition specifications give assurance of satisfactory material. For the lubrication of some machines and accessories, the demands of which are particularly severe, lubricants recommended by the manufacturer of the machinery have been purchased. This system of temporizing has resulted in the growth of an extensive list of proprietary lubricants, with the attendant complication of maintaining stocks of twenty or thirty special lubricants at Naval bases and yards and on supply ships and aircraft carriers. A particularly objectionable feature has been the necessity of stocking several different lubricants for similar machines from different manufacturers.

Desirability of Performance Tests

Government Departments, particularly the Navy, have long favored the purchase of materials on a service life or work-factor basis. In the case of lubricating oils, this method has been so far developed that the work factor is applied to the manufacturer's bid price with the result that the Government instead of purchasing lubricating oil buys lubrication with oil. Work on greases has been in progress at the Engineering Experiment Station for several years and it now appears that the specification of greases on the basis of performance instead of the former specification on the basis of composition and physical properties is possible. The work is by no means completed, but a description of the special apparatus and the results which have been obtained to date should be of interest to both producers and consumers of greases.

The object of these methods is to measure the working lubricating proper-

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[1] The opinions expressed in this paper are those of the authors and are not necessarily official opinions of the Navy Dept. or the Engineering Experiment Station.

[2] Chemical Engineer in Charge of Research Section of Chemical Laboratory, and Contract Employee Engaged in Research on Greases, respectively, U. S. Naval Engineering Experiment Station, Annapolis, Md.

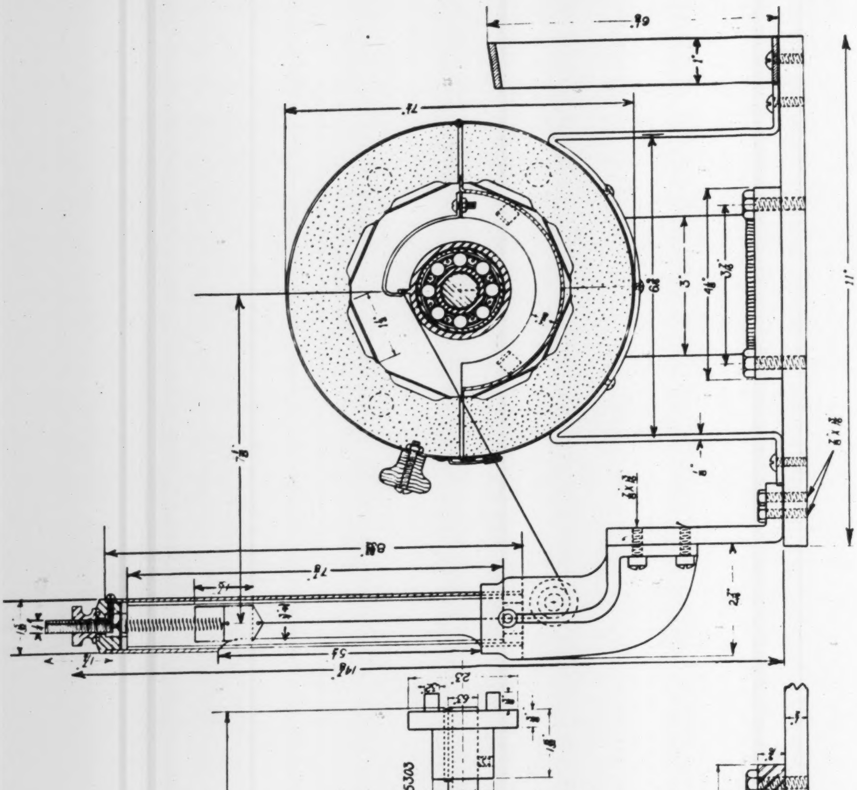
SIDE ELEVATION
FRONT ELEVATION
5303
204-F
Studs
Thermocouple
11"
3"

FIG. 1.—Navy Grease Machine, 1938.

ties of grease without regard to the materials of which it is composed, the manner of manufacture, or its history prior to receipt. While all of these affect its properties, the evaluation of such diverse factors is impracticable. Naturally the first efforts were directed toward lubricants for apparatus in which the greatest difficulty had been encountered.

HIGH-TEMPERATURE GREASE

The first of these was the specification of lubricants for high-temperature service, in situations where grease would be exposed to temperatures of 300 F. or more. An apparatus was designed and built, Navy Grease Machine 1938, Fig. 1, in which a standard ball bearing, filled with the test lubricant, can be operated at controlled temperature and speed, while the torque is measured. All else being equal, it is reasonable to believe that the grease with the lowest torque and therefore the lowest friction loss would be the most suitable. Many precautions are necessary in order to obtain reproducibility. The first is the maintenance of a uniform filling of the test bearing with grease. To accomplish this a filling apparatus, called the Navy Bearing Packer, Fig. 2, was made, with which all voids in a No. 204 open bearing can be filled. The amount of grease loaded into the test bearing by means of the bearing packer is reproducible within 1 per cent.

Principal variables affecting the torque measurements, in addition to the character of the grease, are temperature, amount and distribution of grease in the bearing, speed of rotation, and time since the start of the test. The last is important because of the thixotropy of the grease, since during working its plasticity is constantly changing. Time can be measured, but not altered or reversed. In order to obtain reproducible results, it is necessary to collect all data in the form of torque-time curves. All other variables can be maintained constant throughout the test run.

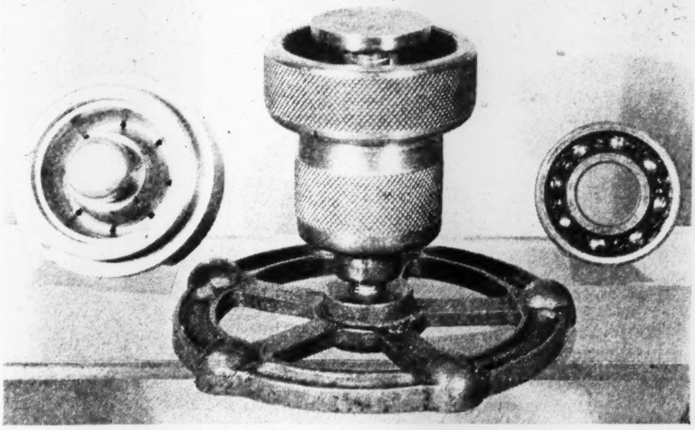

FIG. 2.—Navy Bearing Packer.

At the start of the test run the grease is packed into 100 per cent of the voids of a weighed, No. 204, eight-ball bearing without grease shields, and the filled bearing is again weighed. The outer race of the test bearing is clamped into a brake drum and the assembly locked on the shaft of the Navy Grease Machine with the shaft through the inner race, and the brake drum connected to the torque-measuring spring. The motor previously has been adjusted to rotate at the desired speed and the test temperature is set. The motor is started and the first excursion of the spring is read as the initial torque, with readings thereafter at first frequently, then at increasing intervals, for 1 hr. Although the torque has been observed to decline continuously, at a decreasing rate, for as long as 16 hr., the torque-time curve obtained in 1 hr. is characteristic. At the end of the run the bearing is removed, excess grease is wiped off the faces of the race, and the bearing weighed. The difference in weight is called leakage and is calculated to percentage of the original grease charge.

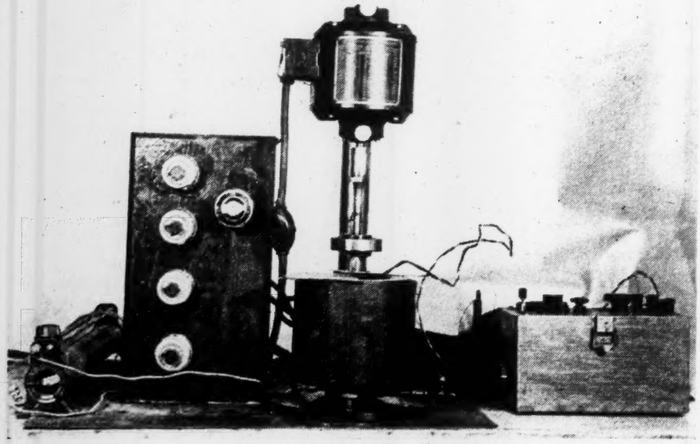

FIG. 3.—High-Temperature Beater.

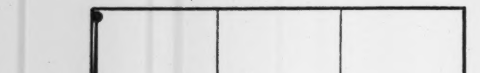

FIG. 4.—Torque Measurement on Grease Before and After High-Temperature Beating.

The deterioration of stable lubricants under high-temperature conditions is not rapid enough to be measured by tests under simulated service conditions in the Navy Grease Machine. In order to accelerate the rate of oxidation and deterioration, a high-temperature beater was devised. This apparatus, as illustrated in Fig. 3, consists of a single-row ball bearing, $2\frac{3}{4}$ in. in outside diameter, with the inner race clamped by a wing nut to the lower end of the vertical shaft of a 3600-rpm. motor. A counterbalanced cup with electrical heaters in its jacket can be raised onto the bearing so that vertical ribs in the interior of the cup hold the outer race and prevent its rotation.

The standard high-temperature stability test consists of placing ½ lb. of grease in the cup, heating to 400 F., and then beating with the rotating bearing for 7 hr. At the end of the test the residue, or grease, if it is still recognizable, can be used to fill a test bearing and repetition of the Navy Grease Machine tests gives an indication of the extent of damage to the original properties of the lubricant. The slight increase in torque after beating indicated in Fig. 4, illustrates a grease satisfactory for high-temperature service.

On the basis of results obtained with the above apparatus, the Engineering Experiment Station has prepared a specification for high-temperature lubricants[3] which has been used for purchases of high-quality greases for almost 2 yr. The results have been entirely satisfactory.

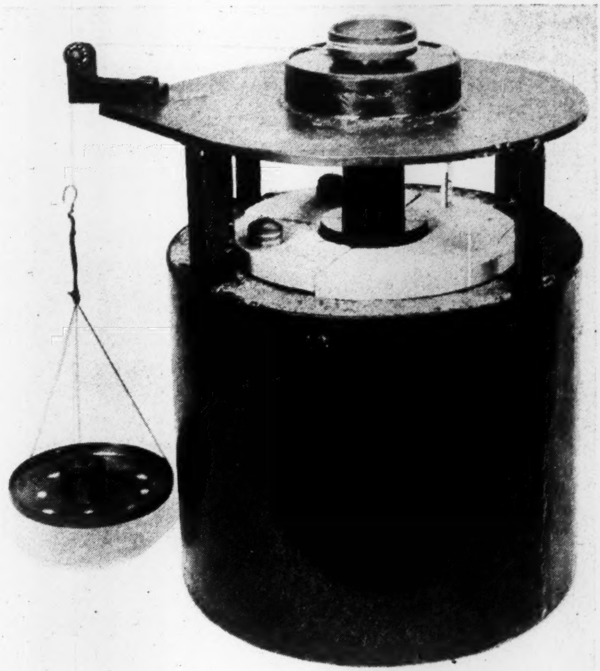

FIG. 5.—Cold-Test Apparatus.

LOW-TEMPERATURE GREASE

The second application of performance testing to specification of greases was to the problem of aircraft-control lubricants. Bearings in aileron and rudder hinges and in pulleys in control cable trains are exposed to ambient temperatures. This means that bearing and lubricant temperatures may vary from that of metal in tropical sunlight, about 165 F., to the temperature of the sub-

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[3] Navy Department Aeronautical Specification M-372, dated September 1, 1939; copies obtainable from Bureau of Aeronautics, Navy Dept., Washington, D. C.

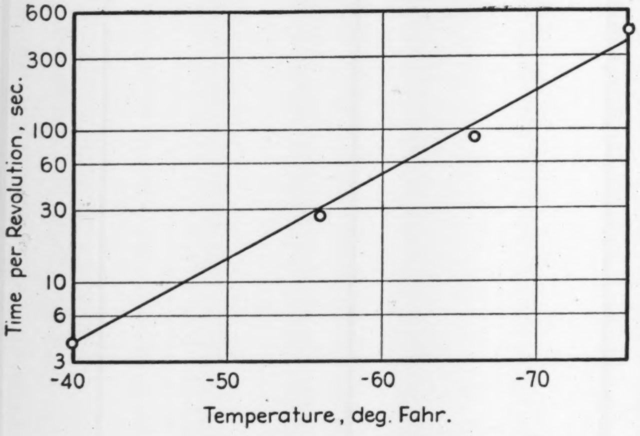

FIG. 6.—Effect of Temperature on Time for Rotation.

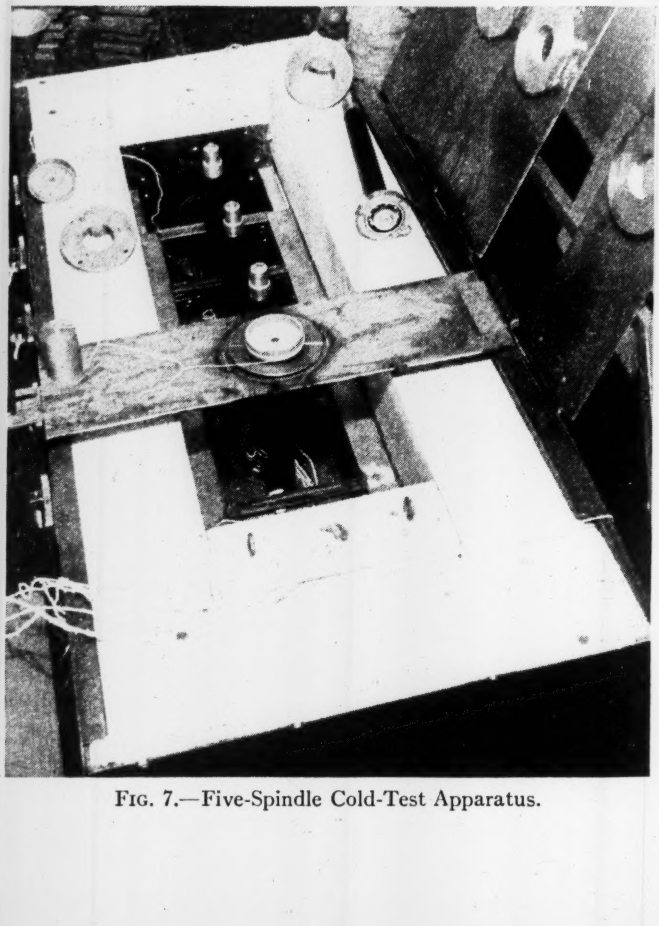

FIG. 7.—Five-Spindle Cold-Test Apparatus.

stratosphere of −60 to −65 F. No one can predict suitability throughout such a range from composition and penetration at 77 F. Performance tests are required.

The original apparatus for low-temperature mobility testing is illustrated in Fig. 5. It consists of a vertical spindle of hard rubber with the inner race of the test bearing clamped to the lower end and a pulley on the upper end. The outer race of the test bearing is held between the two parts of a liquid-tight sleeve so that sleeve and bearing can be placed in a bath of alcohol and dry ice without wetting the lubricant in the test bearing. The spindle has an axial hole for a thermometer whose bulb lies at the center of the test bearing. After the accurately lubricated test bearing has been assembled in the apparatus and cooled to the test temperature (−40 or −50 F.) a predetermined torque is applied through the pulley and the time required for the initial revolution is determined. Temperature is the most critical of the several factors affecting consistency and reproducibility of results. The relationship between plasticity of the grease and temperature is similar to the viscosity-temperature curve for lubricating oil. As shown in Fig. 6, the logarithm of the time of rotation *versus* the temperature gives a straight line.

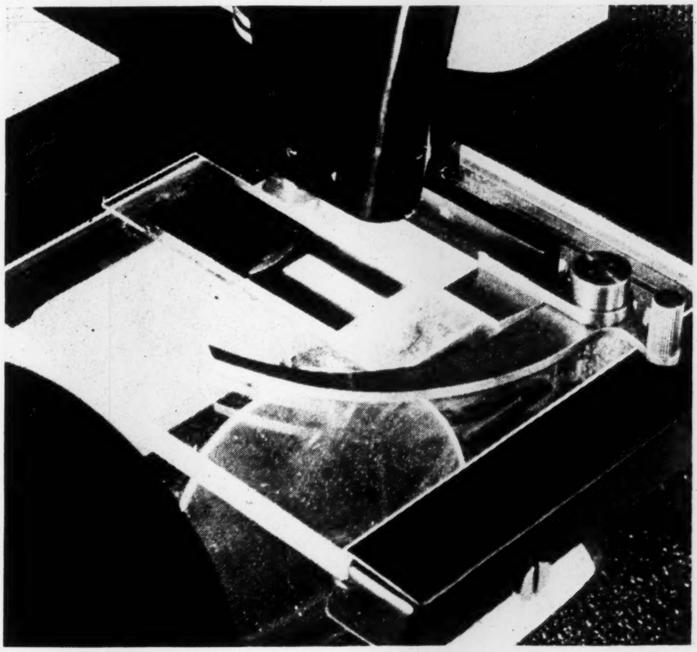

FIG. 8.—Dirt-Count Apparatus.

In order to obtain more accurate temperature control and at the same time to increase the number of determinations per man-day a five-spindle, air-bath apparatus has been built. This is shown in Fig. 7. A rectangular, jacketed, steel trough supports the five spindles. A small pump circulates alcohol from a coil in the cold bath at the bottom through a continuous coil of finned tubing in the jacket, while a centrifugal blower at one end of the trough maintains circulation of air through the jacket and back over the spindles and test bearings. Temperature is controlled by a pyrometer in the pump-motor circuit actuated by a thermocouple in the air stream in the trough. The temperature difference from end to end of the trough does not exceed 2 F. after equilibrium has been attained so that the temperature on any spindle is within 1 F. of the control temperature.

In addition to plasticity at low temperature, a suitable lubricant must be stable at the highest temperature to which it will be exposed. There are two ways in which a grease may fail when heated; it may change from a plastic structure to a fluid and run out of the bearing (leakage), or it may separate into a stiffer plastic by losing some of the oil (bleeding). To test for leakage, the bearing which has been used for the cold test is run in the Navy Grease Machine 1938 for 1 hr. at 150 F. and 600 rpm. Not more than 5 per cent of the original charge should leak from the bearing during this test. For separation or bleeding tests, 10 g. of grease are weighed into a perforated nickel filter cone which is exposed in a tared 100-ml. beaker and placed in a drying oven, kept at 150 F. During 50 hr. no oil or molten grease should drip into the beaker. The quantity of such leakage is determined by weighing the beaker with its separated product.

DIRT IN GREASE

The application of grease lubrication to high-speed precision bearings having minimum clearances makes necessary control of the non lubricant inclusions in grease. Such things as sand, uncombined lime, metal particles, hair, and vegetable fibers are present in all greases. In most applications this dirt causes no trouble; at least no casualties have been attributed to it. It seems reasonable, however, that serious damage might be done to a ball bearing if a particle of sand having a diameter twice the clearance between balls and races were thrown into the ball track when the bearing was rotating at high speed. In order to establish standards of cleanliness a quantitative measure of the dirt in grease must be possible.

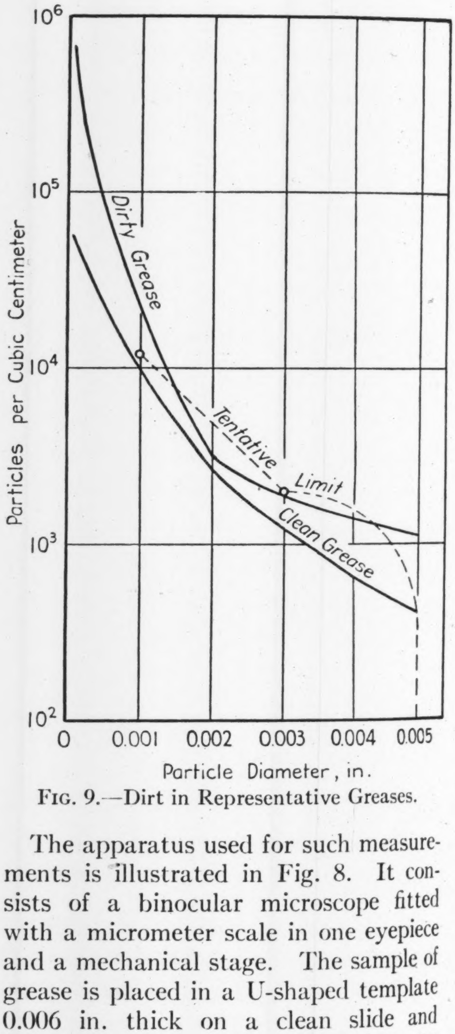

FIG. 9.—Dirt in Representative Greases.

The apparatus used for such measurements is illustrated in Fig. 8. It consists of a binocular microscope fitted with a micrometer scale in one eyepiece and a mechanical stage. The sample of grease is placed in a U-shaped template 0.006 in. thick on a clean slide and exposed to a 29 to 30 in. vacuum for 10 min. after which a cover glass is placed over the sample. Exposure to vacuum removes entrained air bubbles and clarifies the sample for examination. Magnification of 60 diameters has been found most usable. The sample is illuminated simultaneously from below and transversely. The slide bearing the sample is placed on the stage of the microscope so that the base and sides of the template are parallel to the two motions of the mechanical stage and the eyepiece scale is turned until parallel with the sides of the template and with one end in a corner of the sample. The sample is then racked across the field of view while the observer counts the particles as they pass the eyepiece scale. The individual particles can be measured and their sizes recorded as they are counted.

The results of examination of two greases are illustrated in the curves of Fig. 9. Each ordinate of these curves represents the concentration of particles of the indicated size or larger which were present in the sample. Particles smaller than 0.0005 in. in diameter were counted at a magnification of 113 diameters. Both greases were quality products of their respective manufacturers and the cleaner of the two is among the cleanest which has been examined. The absence of particles larger than 0.005 in. in diameter should be secured by adequate screening of the finished grease but low concentrations of foreign material smaller than one or two thousandths of an inch probably can be obtained only by care in the selection of raw materials and cleanliness of equipment during manufacture. A tentative limit of foreign matter in commercially obtainable greases is indicated by the broken line in Fig. 9: namely,

Minimum Diameter, in.	Particles per Cubic Centimeter Above Minimun Diameter
0.001	12 000
0.003	2 000
0.005	0

ADVANTAGES OF PERFORMANCE SPECIFICATIONS

The use of performance specifications, obtained by coordination of results from special test equipment, has resulted in the purchase of definitely superior lubricants. Previously such materials were obtainable only by proprietary purchase. In the light of these test procedures the requisite properties for a satisfactory lubricant for a specific troublesome application can be visualized and tentative requirements can be outlined, the available lubricants can be tested and their efficiency observed. Cooperation between the testing station and the manufacturer often serves to remedy deficiencies with the result that the manufacturer has a premium product to market and the purchaser can obtain a lubricant which gives satisfactory service with a net reduction in the maintenance cost.

A correlary discovery resulting from this investigation is the economy which may result from the use of superior greases. The special testing procedures were developed for specification and purchase of satisfactory greases for difficult applications. Lubricants having the extraordinary properties demanded by these performance specifications are supplied at considerably higher prices than the greases meeting the old composition specifications. Despite this higher unit price, lubrication with these superior lubricants may be more economical than with ordinary lubricants.

Chassis lubrication of a passenger automobile or light truck may be used as a demonstration. Lubrication for such a vehicle at 1000-mile intervals for an estimated 15,000 miles per year would cost $16.20 per annum, with labor at one dollar per lubrication and grease at eight cents per pound. We have found that chassis lubrication with the medium grade of grease supplied under Navy Aeronautical Specification M-372[3] is required at only 7000- to 8000-mile intervals. This grease is heavier in consistency than ordinary chassis lubricants so that the labor cost of lubrication with it is greater than with ordinary semifluid chassis lubricants. Even if the labor cost were twice as great, (two dollars per lubrication), the annual cost would be only four dollars, and the grease could cost as much as seventy cents per pound and still provide lubrication for one third the cost of ordinary lubrication.

In the applications for which performance specifications were developed, monetary considerations are relatively unimportant. Less than 1 lb. of grease will be required for all control bearings of a large bomber or transport plane. The primary consideration is not whether the pound of grease costs ten cents or ten dollars but whether it will provide satisfactory lubrication under all service conditions or will freeze and make the controls inoperable at high altitudes and low temperatures thus hazarding the entire plane with its more precious crew and mission.

SUMMARY

The purpose of performance specifications is to obtain the most satisfactory and economical grease for each application regardless of the materials or method used in its manufacture. They should free the manufacturer from arbitrary restrictions in his choice of method and materials, simultaneously opening to him the opportunity for recognition of truly superior products. The beginning which has been made offers promise of universal use to the mutual advantage of both producers and consumers of grease.

DISCUSSION

Mr. C. L. Knopf.[1]—I should like to ask why the authors run their test at 400 F. Is this supposed to be a service condition?

Mr. Robert C. Adams.[2]—We do not consider 400 F. representative of service conditions since we believe that no petroleum product can withstand extended service at 400 F. or above. However, on this accelerated test, completed within a reasonable time, we accelerate by increasing the temperature to 400 F.

Mr. Knopf.—Could you not accomplish the same thing by operating at a lower temperature for a longer time? Do you feel that your test is comparable to service conditions when you raise the temperature and speed for a short time? In other words, is it your idea to oxidize the grease?

Mr. Adams.—That is right.

Mr. Knopf.—How thoroughly do you oxidize it?

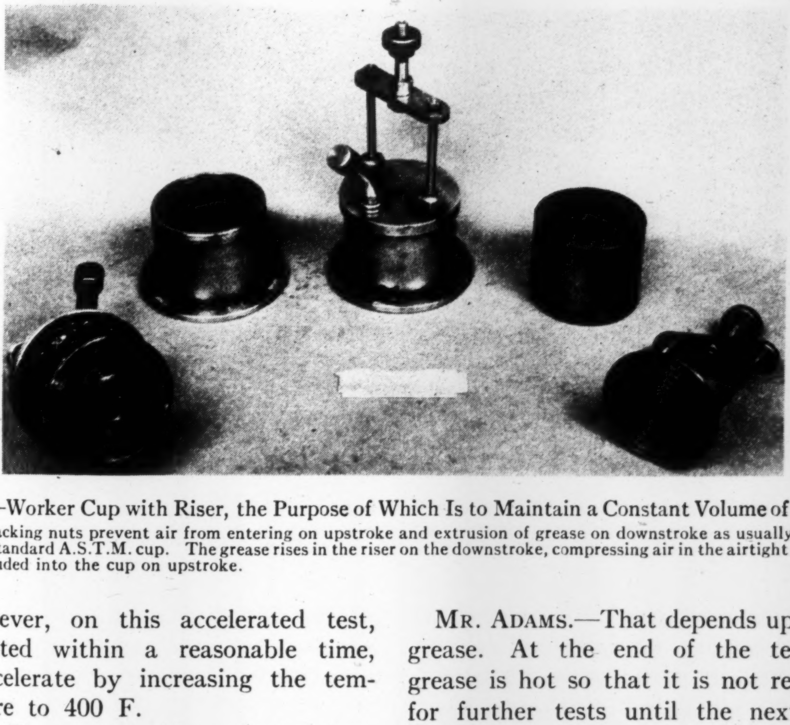

Fig. 1.—Worker Cup with Riser, the Purpose of Which Is to Maintain a Constant Volume of Grease.

The packing nuts prevent air from entering on upstroke and extrusion of grease on downstroke as usually happens with the standard A.S.T.M. cup. The grease rises in the riser on the downstroke, compressing air in the airtight chamber. It is extruded into the cup on upstroke.

Mr. Adams.—That depends upon the grease. At the end of the test the grease is hot so that it is not removed for further tests until the next day. Some greases have to be chiseled out, and others are virtually in the same condition they were at the start.

Mr. Knopf.—Your method circulates the grease and oxidizes it?

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[1] Staff Engineer, Sinclair Refining Co., New York, N. Y.

[2] Chemical Engineer in Charge of Research Section of Chemical Laboratory, U. S. Naval Engineering Experiment Station, Annapolis, Md.

Mr. Adams.—Yes. The nut which retains the bearing which is immersed in the grease, is in the form of a wing nut and that acts as a propeller. During the beating, the grease circulates downward through the ball race and up and around the outer race of the bearing. It is very thoroughly mixed during all that time.

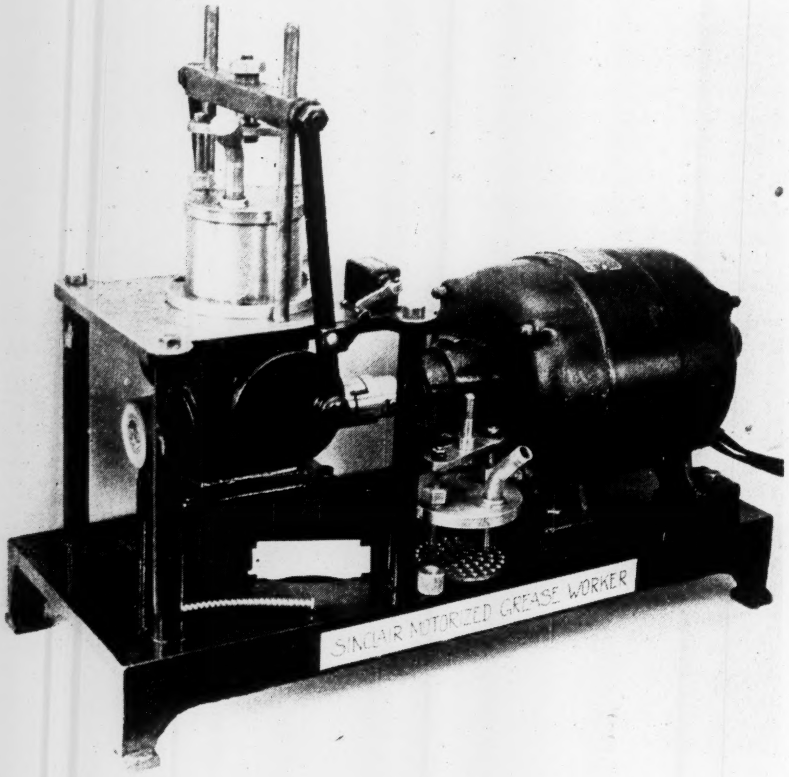

Fig. 2.—The Motorized Worker Developed 10 or 12 yr. Ago for the Purpose of Studying the Stability of Greases.

We sometimes have to work a grease (a stable product) 30,000 strokes before it is down to an ultimate consistency. Consistencies are measured by means of the Consadometer, reading in grams force required to deform the grease. Plunger head, 1.3 in. in diameter.

Mr. Knopf.—A motorized mixer, which we developed 10 or 12 yr. ago, has been used recently to work greases in an A.S.T.M. cup which is provided with a heating element, and a means for introducing air during each downstroke (see accompanying Figs. 1–4). Greases, worked 10,000 strokes ($2\frac{3}{4}$ hr.) at 250 or 300 F., are so oxidized that final Consadometer consistencies are many times the original.

Messrs. Adams and Harrison E. Patten[3] (*authors' closure, by letter*).—The authors do not consider that the apparatus and methods described cannot be improved. The same results probably could be obtained with entirely different apparatus, as suggested by

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[3] Contract Employee Engaged in Research on Greases, U. S. Naval Engineering Experiment Station, Annapolis, Md.

Mr. Knopf. What we consider to be new and distinctive in our development is the incorporation of the results of objective, performance tests in purchase specifications. Instead of writing a prescription which tells a manufacturer how he shall make his product, the Navy now can specify what it requires a grease to do and, in so specifying, can demand the best that the most competent manufacturer can supply.

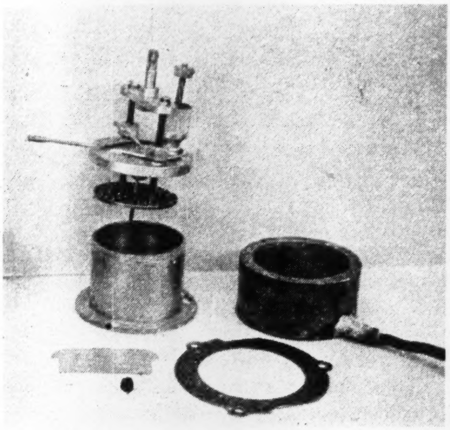

FIG. 3.—The A.S.T.M. Mixer Cup with Cover Suspended Above.

The air inlet tube is at left. Thermocouple tube is in the middle of the cup. The poppet vent valve is on the right side of the lid. The vent valve opens at top of stroke and closes at bottom, being open during entire downstroke. The air is admitted at the top of the stroke and closed $\frac{5}{8}$ downstroke; the remaining part of stroke is employed to allow pressure to drop to atmospheric while the vent is still open. Heating element is at right.

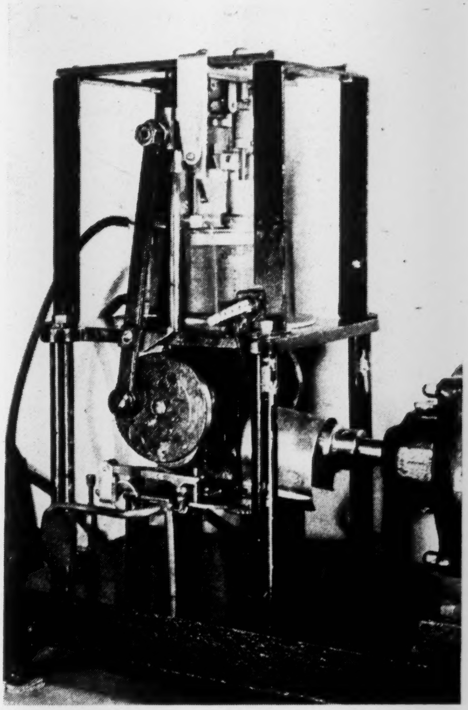

FIG. 4.—Vent Valve Mechanism (Shown at Top).

Timing valve is operated by cam. Air flow meters and pressure regulators are also employed, but not shown.

FUNDAMENTAL SIGNIFICANCE OF OLIENSIS SPOT TEST—QUANTITATIVE TESTS FOR HOMOGENEITY

By G. L. Oliensis[1]

Synopsis

Hitherto it has been claimed for the spot test that it can detect (1) cracked or overheated asphalt; (2) asphalt containing waxy or other bodies that make it especially susceptible to moderate refining temperatures; and (3) asphalt containing sludge or other bodies that are for any reason poorly tolerated by the asphalt system, irrespective of the refining temperatures used.

All the foregoing three claims can logically be condensed into the following single broad principle: That the spot test reacts "negative" with all asphalts in which the internal phase relationship is relatively stable, so that the latter can withstand successfully the standardized flocculating force exerted by the conditions of the test; and reacts "positive" with all asphalts in which the internal phase relationship is relatively unstable (from whatever cause—whether overheating, or the presence of naturally present or artificially added incompatible bodies), so that it is incapable of withstanding that flocculating force.

Sporadic cases that have been cited as apparently inconsistent with any of the first three narrower claims, are shown to be consistent with this broad principle underlying each of them.

To supplement the xylene equivalent previously released for the quantitative determination of heterogeneity, two alternate methods for the quantitative determination of homogeneity are proposed: One, the "negative period," being the number of days the standard naphtha-asphalt mixture can stand before becoming "positive"; the other, the "hexane resistance," being based upon the minimum number of increments of hexane required to make a "negative" standard naphtha-asphalt mixture test "positive." These quantitative methods will aid in detecting in "negative" asphalts (*a*) very slight degrees of overheating or cracking, and (*b*) the presence of small added percentages of "positive" asphalts.

In 1933[2] the spot test was first proposed for a single concrete function: (1) to identify cracked and overheated asphalt. By 1936[3] it had been found that the spot test can also identify (2) asphalt containing waxy or other poorly tolerated bodies that make it abnormally susceptible to relatively low refining temperatures; and (3) asphalts containing sludge, weathered, or any other bodies (either naturally present or artificially added)[4] that are for *any* reason poorly tolerated by the asphalt system, irrespective of the refining temperatures employed.

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[1] Chemist in Charge, Technical Dept., Barber Asphalt Corp., Madison, Ill.

[2] G. L. Oliensis, "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 715 (1933).

[3] G. L. Oliensis, "A Further Study of the Heterogeneity of Asphalt—a Quantitative Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 494 (1936).

[4] By "artificially added" bodies are meant, for example, those resulting when separate fractions are recombined (as in "synthetic asphalts") in such a way and in such proportions as to become mutually incompatible.

A close analysis of the three claims advanced for the spot test discloses that each of them, while originally formulated independently of one another and as the result of separate phases of study, is logically merely an amplification or outgrowth of its predecessor, each approaching progressively closer to the true and ultimate scope of the test, which is wider than each of the claims separately and is inclusive of them all.

This wider interpretation, which is most nearly foreshadowed in the third claim, is based to some extent on the generally accepted conception that all asphalts are mixtures of at least three groups of hydrocarbons—petrolenes, resins, and asphaltenes; but a probably truer conception of asphalt is that it is a mixture of a practically unlimited number of phases merging imperceptibly into one another and held in a more or less stable mutual solution, or more correctly, in a more or less stable colloidal or semicolloidal suspension. In the spot test a relatively large volume of relatively light hydrocarbon (heavy naphtha) is purposely added to weaken the surface tension of the lighter phases, thereby increasing the interfacial tension between them and the heavier phases, and impairing their mutual solubility. The degree of this impairment exerted by the conditions of the test, is arbitrary, but is carefully and rigidly controlled so as to be uniform in all cases.

In the normal or "relatively homogeneous" asphalt—for example, vacuum or steam refined residuals from a given crude, refined at normal temperature—the mutual solubility of the various lighter and heavier phases in one another (that is, the "phase relationship") is so stable that it cannot be disturbed or destroyed by this dilution with the prescribed amount of the standard naphtha; hence no insoluble dispersion develops.

However, in the "relatively heterogeneous" asphalt—for example, overheated or cracked residuals (in which the overheating has modified one or more phases to the extent where they become less compatible with the system than in the case of the normal asphalt), or in asphalts containing paraffin, or sludge, or weathered or other bodies, either naturally present or artificially added[4] (which either become incompatible because abnormally susceptible to relatively low refining temperatures, or are inherently incompatible "as is")—the stability of the phase relationship has through the presence of these bodies become sufficiently disturbed as to render the system incapable of withstanding the flocculating force exerted by the conditions of the test; hence precipitation results.

According to this broad principle, the fundamental function of the spot test is to determine, not whether an asphalt is cracked, weathered, or improperly synthesized, but primarily whether its internal phase relationship is relatively stable (as indicated by a "negative" spot) or relatively unstable (as indicated by a "positive" spot). It is true that the spot test has been found very useful in detecting cracked or overheated asphalts; but that has been solely because the latter happen to be the only common type on the market whose internal phase relationship is relatively unstable (under the certain standard conditions of the test as to dilution with a specified diluent), the other relatively unstable types being much rarer.

To the class characterized by a relatively stable internal phase relationship (that is, "relative homogeneity") belong the normally refined and all other asphalts in which all constituent phases or bodies are mutually compatible. To the class characterized by a relatively unstable internal phase relationship (that is, "relative heterogeneity") belong all asphalts that contain relatively incompatible bodies of one or more of the following types:

1. Bodies that through cracking or overheating "get out of phase" (that is, become incompatible) with the other phases of the asphalt.
2. Bodies whose potential incompatibility renders them abnormally susceptible to relatively moderate heating—for example, certain waxy bodies.
3. Bodies that have become incompatible through causes other than heating—for example, weathering.
4. Bodies that are *inherently* incompatible with the asphalt system—for example, some constituents of sludge or coal-tar pitch.
5. Bodies that are not inherently incompatible, but become so through faulty synthesis or proportioning of the component fractions.

Once the spot test, by virtue of its classifying or identifying functions, has established a positive reaction, it may then be desired to determine to which of the foregoing five subdivisions it is due; that is, whether the unstable phase relationship of the asphalt under consideration is due to its having been cracked, or weathered, or improperly synthesized; but such supplementary determinations are manifestly beyond the scope of the standard spot test.

From time to time specific cases have come to the author's attention that have appeared to challenge the foregoing claims as to the scope of the spot test. For example, it has been stated:

1. That occasionally asphalts that have never been overheated in refining nevertheless react "positive." (Several asphalts of this type, cited in a paper by Born,[5] were in my discussion of that paper shown to be characterized by an incompatibility of the "waxy bodies" type, vulnerable to even moderate refining temperatures.)

2. That occasionally asphalts that have been slightly overheated nevertheless react "negative" to the spot test. (These asphalts have been discussed in detail by the author elsewhere.[6]) In that discussion it was shown that (*a*) cracked asphalts and all asphalts that had been overheated in the ordinary sense of the term, tested "positive" in all cases; (*b*) a very slight degree of overheating might not be detected by the standard method for making the spot test; but (*c*) even such cases of slight overheating could be detected by a refinement of the spot test, based upon the tendency of asphalts to develop heterogeneity in naphtha solution the more quickly, the greater the degree of overheating to which they had been subjected during manufacture. (Incidentally, this tendency forms the basis of the quantitative determination of the "negative period" to be discussed later in this paper.)

3. It has also been stated that when fractionating asphalts into petrolenes, resins and asphaltenes, and then recombining the latter in varying proportions, (*a*) fractions derived from uncracked asphalts may be occasionally recombined in such a way as to yield a "positive" spot, even though none but the lowest temperatures necessary to blend them had been used;[7] and (*b*) fractions derived from cracked asphalts may be recombined occasionally in such a way as to yield a "negative" spot.

Though on the face of it the foregoing

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[5] Sidney Born, "The Homogeneity of West Texas Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 519 (1937).

[6] G. L. Oliensis, "Efficacy of Oliensis Spot Test on Slightly Cracked Asphalts," *Proceedings*, Montana National Bituminous Conference, p. 193 (1939).

[7] J. Zapata, "Notes on the Composition and Testing of Asphalts of the Slow Curing Type," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 10, January, 1939, p. 160.

appears inconsistent with the claims advanced for the spot test, yet when we bear in mind the third of the three claims in the first paragraph of this paper, and more particularly the broad principle that has been shown to underlie them all, it appears entirely logical that when recombining separate phases or fractions, such fractions, even though derived from a normal or "negative" asphalt, may nevertheless in some instances be regrouped in such a way as to decrease the mutual solubility of all phases so as to disturb measurably the stability of the phase relationship of the resultant blend. In fact, not only may it thus be disturbed enough to develop precipitation upon the addition of the standard naphtha, but in extreme cases we know that it may thus be destroyed entirely, so that copious precipitation or sludging may result even before the addition of any naphtha whatsoever.

Conversely, it should be possible in some instances to recombine fractions from an overheated or "positive" asphalt in such carefully balanced proportions, as to improve the stability of the phase relationship to such a point that it can no longer be disturbed, far less destroyed, by the later addition of the standard naphtha. Hence the synthesis of a "positive" asphalt from fractions derived from a "negative" asphalt, or the synthesis of a "negative" asphalt from fractions derived from a "positive" one, while an unusual phenomenon, is not necessarily inconsistent with the generally accepted understanding of the scope of the spot test.

In brief, in the various sporadic cases cited as apparently contradicting any of the three narrower claims heretofore made for the spot test, a careful analysis will disclose that they are entirely consistent with the broad principle underlying each of them as above explained; for the basic function of the spot test is to distinguish between asphalts whose internal phase relationship is either stable or unstable to the dilution and the diluent specified in the method, irrespective of how such stability or instability may have been brought about.[8]

NEED FOR QUANTITATIVE TEST FOR HOMOGENEITY

Nevertheless, while the primary function of the spot test is this qualitative one of dividing all petroleum asphalts into two main categories, it has always been apparent that such a test would have a far larger sphere of usefulness if it were implemented so as to be also quantitative in scope. With that in mind, the author, in 1936, developed the "xylene equivalent"[3] as a method for determining quantitatively the degree of heterogeneity of such asphalts as were positive to the spot test. To develop a corresponding method for determining the relative degree of homogeneity of negative-spot asphalts proved a more difficult task. The ideal solvent for such a method would, of course, have been some hydrocarbon of at least as pure chemical composition as the xylene used in the xylene equivalent, but which would as completely flocculate every petroleum asphalt as xylene completely dissolved it. No such pure hydrocarbon, however, appeared available except at prohibitively high prices or in extremely limited quantities.

In 1939 the author reported[6] some work he had done with a number of Wyoming crude-oil residuals (prepared by the Federal Bureau of Mines' Petroleum Experiment Station at Laramie, Wyo.), which revealed that when some of a group of residuals from a given crude oil had been subjected to greater overheating than the others, the spot-test mixtures of the former would on standing invariably become positive more quickly than those of the latter. Since the number of days required to become positive appeared proportional to the degree of overheating, that period offered itself as at least one quantitative measure of the degree of homogeneity of an asphalt. The procedure subsequently worked out follows:

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[8] What practical purpose is served by this broad differentiation will be discussed in the "Conclusions."

TABLE I.—QUANTITATIVE HOMOGENEITY TESTS.

(*A*) STRAIGHT REDUCED RESIDUALS.

Item	Symbol in Figs. 1 to 4	Crude Oil Source	Refinery	Age of Sample	Penetration at 77 F.	Insoluble in 86° Naphtha, per cent[a]	Fixed Carbon,[a] per cent	Negative Period,[c] days	Hexane Resistance, Hexane increments
1	S	Arkansas (Smackover)	1	Recent	45	20.3	(16)[a]	120+[c]	30 or 31
		Arkansas (Urbana)							
2	U(1)	Sli. overheated	1	Recent	76	14.7		58+	20
3	U(2)	Sli. overheated (?)	1	Recent	87	14.5		58+	22
4	U(3)	Normal	1	Recent	88	14.5		58+	36
5	C(1)	California	1	6 yr.	Soft	(8)	(6)	7	9
6	C(2)	California	2	Recent	55	(11)	(8)	72+	37
7	K	Kansas	1	Recent	45	21.0	(14)	72+	51
8		Kansas	1	Recent	150	16.5	(12)	72+	61
9		Kansas	2	2 mo.	52			78+	41
10		Kansas	2	1 wk.	50	25.0	(15)	78+	43
11		Kansas (blend)	2	1 mo.	43	24.3	(15)	78+	30
12	M	Mexican	1	14 yr.	45	(29)	(15)	67	12
13		Mexican	1	14 yr.	175	(26)	(14)	102+	18
14		Mexican	2	9 yr.	50	(29)	(15)	(89 to) 102	18
15		Mexican	2	4 yr.	150	(26)	(14)	102+	22
16		Texas (Hendricks)	1	4 yr.	45	(19)	(14)	85+	29
17			1	Recent	90	(16)	(13)	81+	34
18		Trinidad Oil Resid.	1	Recent	55	12.4	8.3	86+	23
19			1	Recent	155	10.7	6.9	86+	27
20		Venezuela	1	11 yr.	30	(25)	(14)	2 to 3	7
21			2	Recent	55	22.8	13.5	25	16
22	V(1)		2	Recent	155	19.1	11.5	25	16
23	V(2)		3	Recent	85	(22)	(13)	78+	36
24	W(1a)	Wyoming (overheated)	1	2 yr.	325		19.0[b]	2 to 3	7
25	W(1b)	Wyoming (normal)	1	2 yr.	208	(24)	18.2[b]	(65 to) 72	14
26		Wyoming (normal)	1	2 yr.	37	(25)	22.8[b]	96	16
27		Wyoming	2	Recent	45	24.4	(15)	34	14
28			2	Recent	155	22.3	(14)	31	14
29	SY	"Synthetic"	1	Recent	85			79+	22

(*B*) LABORATORY BLENDS WITH CRACKING-COIL TARS.

Item	Symbol in Fig. 4	Smackover, "S" (Item 1), per cent	Cracking-Coil Tar, per cent		Negative Period, days	Hexane Resistance, Hexane increments
			No. 1	No. 2		
30	S + 5% Cr. 1	95	5		106+	23
31	S + 10% Cr. 1	90	10		(73 to) 83	18
32	S + 20% Cr. 1	80	20		3	7 or 8
33		75	25		0 to 1	
34		95		5	93+	27 or 28
35		90		10	93+	24
36		80		20	(49 to) 56	17
37		70		30	9	..
38		65		35	3	..
39		60		40	0	0

[a] Naphtha insoluble and fixed carbon figures in parenthesis are estimated by the author from past data. Those not in parenthesis were furnished by the producers.

[b] Carbon residue.

[c] Negative period figures followed by "+" indicate that the reaction is still negative for the period shown, and is therefore incomplete.

QUANTITATIVE METHOD FOR HOMOGENEITY—NEGATIVE PERIOD

If the mixture of asphalt and standard naphtha prepared for the standard spot test reacts negative at the end of 24 hr., it is set aside in a tightly-stoppered flask in a dark closet, at a temperature maintained uniformly at 77 ± 3 F. (25 ± 1.7 C.). Daily thereafter for three weeks, then thrice weekly for a further three weeks, twice weekly for a further six weeks, and weekly thereafter till the end of the test, it is vigorously swirled for 5 sec. and, after a drop is withdrawn for the spot test, is replaced in the closet. This procedure is repeated at the end of each of the intervals just stipulated, till a positive spot is obtained. If the positive nucleus or ring is very faint, additional spots are obtained at the end of at least two more consecutive intervals, as here scheduled, to make certain that the nucleus becomes progressively more marked, thus demonstrating that its first appearance was authentic and significant. The number of days elapsed between the preparation of the asphalt-naphtha mixture and the appearance of the first *authentic* positive spot, however faint, is called the "negative period."[9]

The negative period for some of the shorter-period negative-spot residuals so far tested in connection with this particular investigation, is found in the next to the last column of Table I. The negative period for the other residuals listed in that table is being determined, and has already extended in some cases to upwards of 120 days, but has not yet been completed. Illustrations of the spots obtained on some of the asphalts in the course of their negative-period determinations are shown at the foot of Figs. 1, 2, 3, and 4, and will be explained more fully later.

Repeated negative-period determinations on the same asphalt yield concordant results if the temperature and time-interval conditions of the test are followed. Higher temperatures than 77 F. (25 C.) substantially reduce the negative period, and lower temperatures substantially increase it. Overheating of the asphalt, or the addition of cracked, overheated, or other positive-spot residuals decrease the period proportionally, as can be noted from Table I (compare item 24 with 25 and 26; item 1 with 30 to 33; item 1 with 34 to 39.)

The negative period would appear to be the simplest and most direct means of measuring homogeneity quantitatively, for it is nothing but a time-amplification of the standard spot test, and requires practically no further laboratory work than is involved in running the standard qualitative determination, except for the moment or two required at the end of the stipulated intervals for the reexamination of the asphalt-naphtha mixture. However, since the negative period will vary with the temperature, and not all laboratories are equipped to maintain the mixtures uniformly at the prescribed temperature, and since the total time required for making this determination may run into many months, the negative-period determination is from present indications better adapted as a research tool than for routine and plant control work.

HEXANE AS AN AID IN MEASURING HOMOGENEITY

On June 13, 1940, Messrs. Winterkorn and Eckert, of the College of Engineering, University of Missouri, were kind enough to submit to the author an advance copy of a yet unpublished paper

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[9] If the development of the positive nucleus in the spot is so gradual that it is difficult to decide just which spot should be considered the first authentic positive one, the author reports the first doubtful one in parenthesis, followed by the first clearly indicated one, thus: "(84 to) 98."

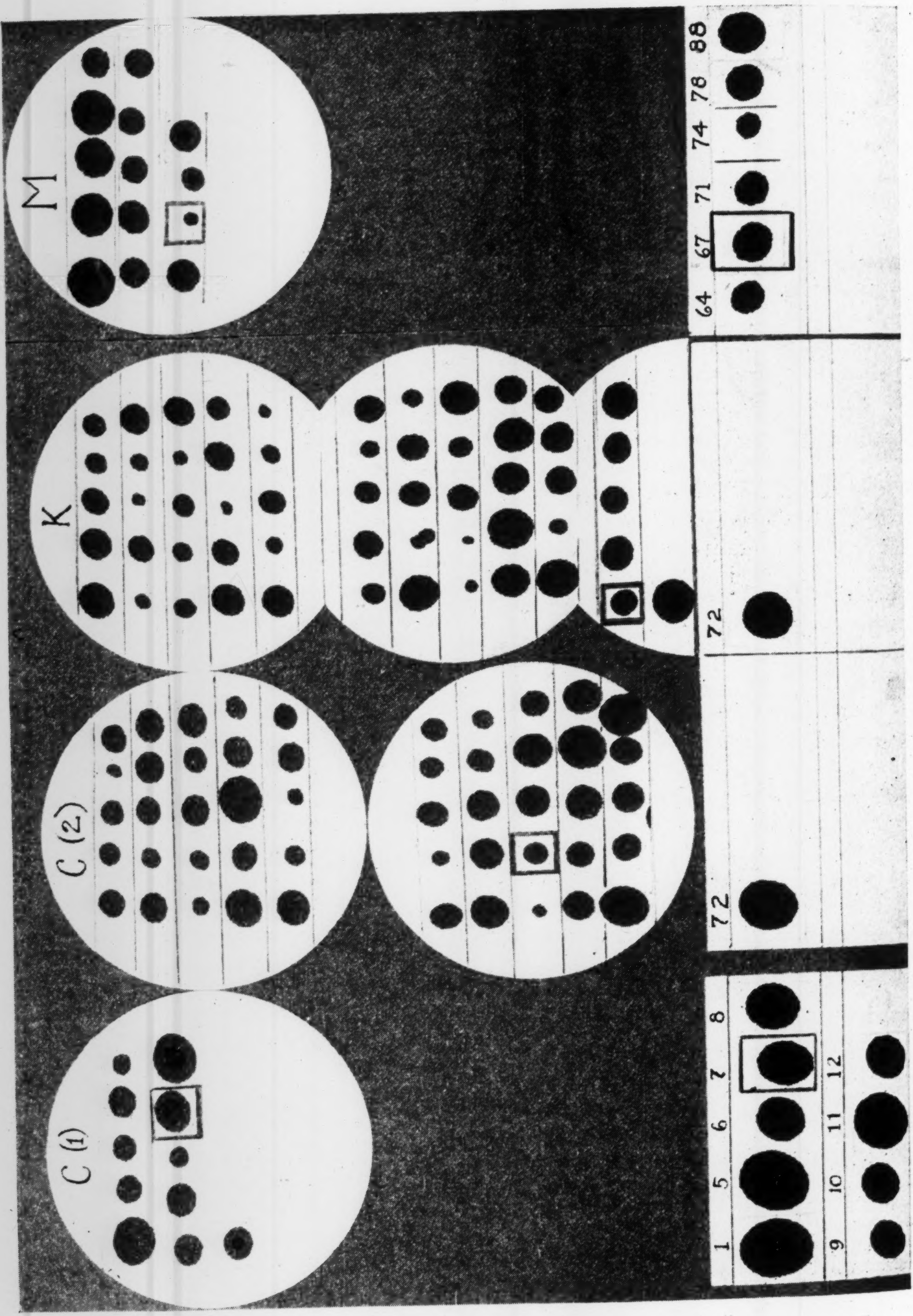
C (1)
C (2)
K
M
1
5
6
7
8
9
10
11
12
72
72
64
67
71
74
78
88

FIG. 1.—Quantitative Homogeneity Tests on California, Kansas and Mexican Residuals.

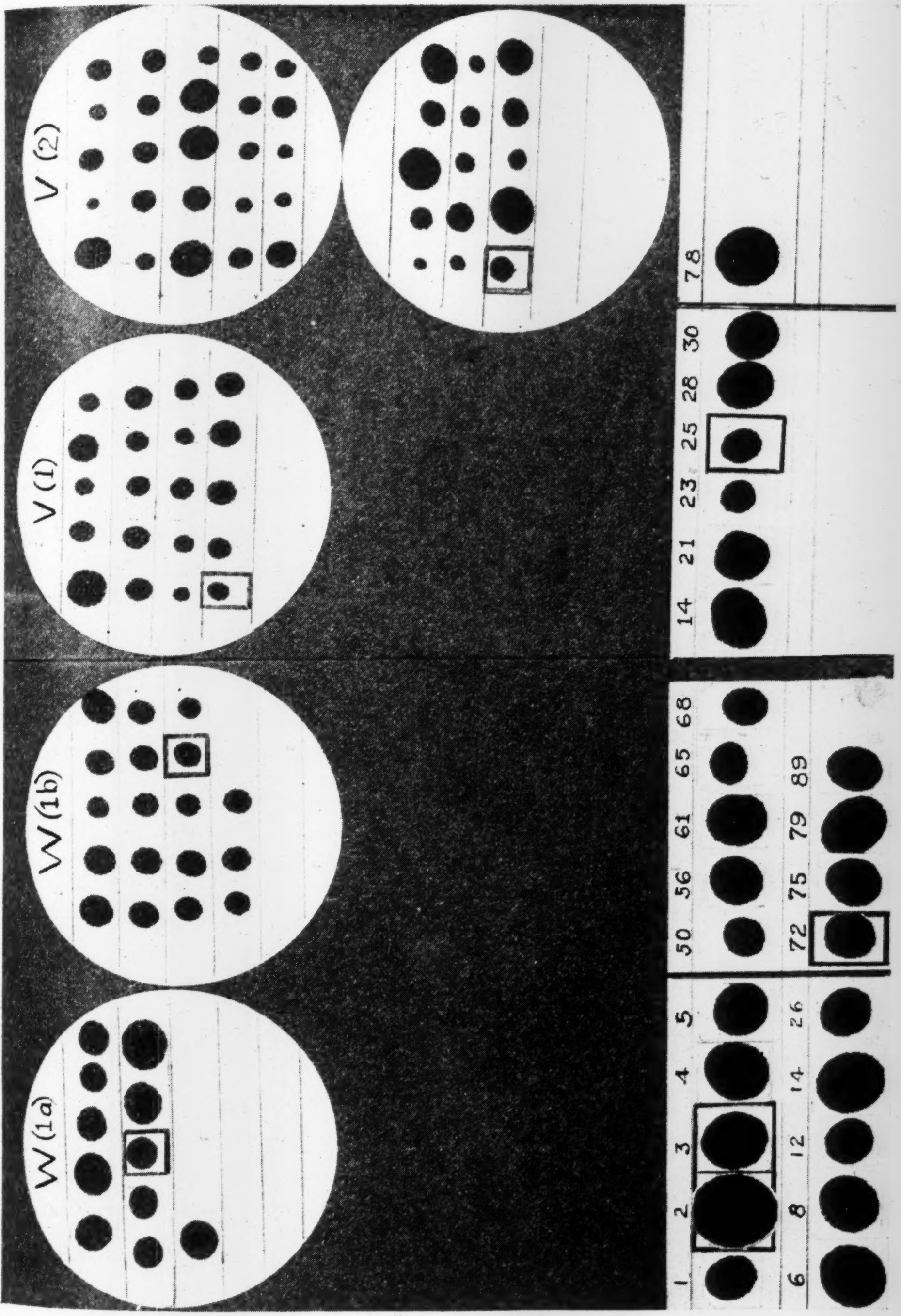
W(1a)
W(1b)
V(1)
V(2)
1
2
3
4
5
6
8
12
14
26
50
56
61
65
68
72
75
79
89
14
21
23
25
28
30
78

FIG. 2.—Quantitative Homogeneity Tests on Wyoming and Venezuelan Residuals.

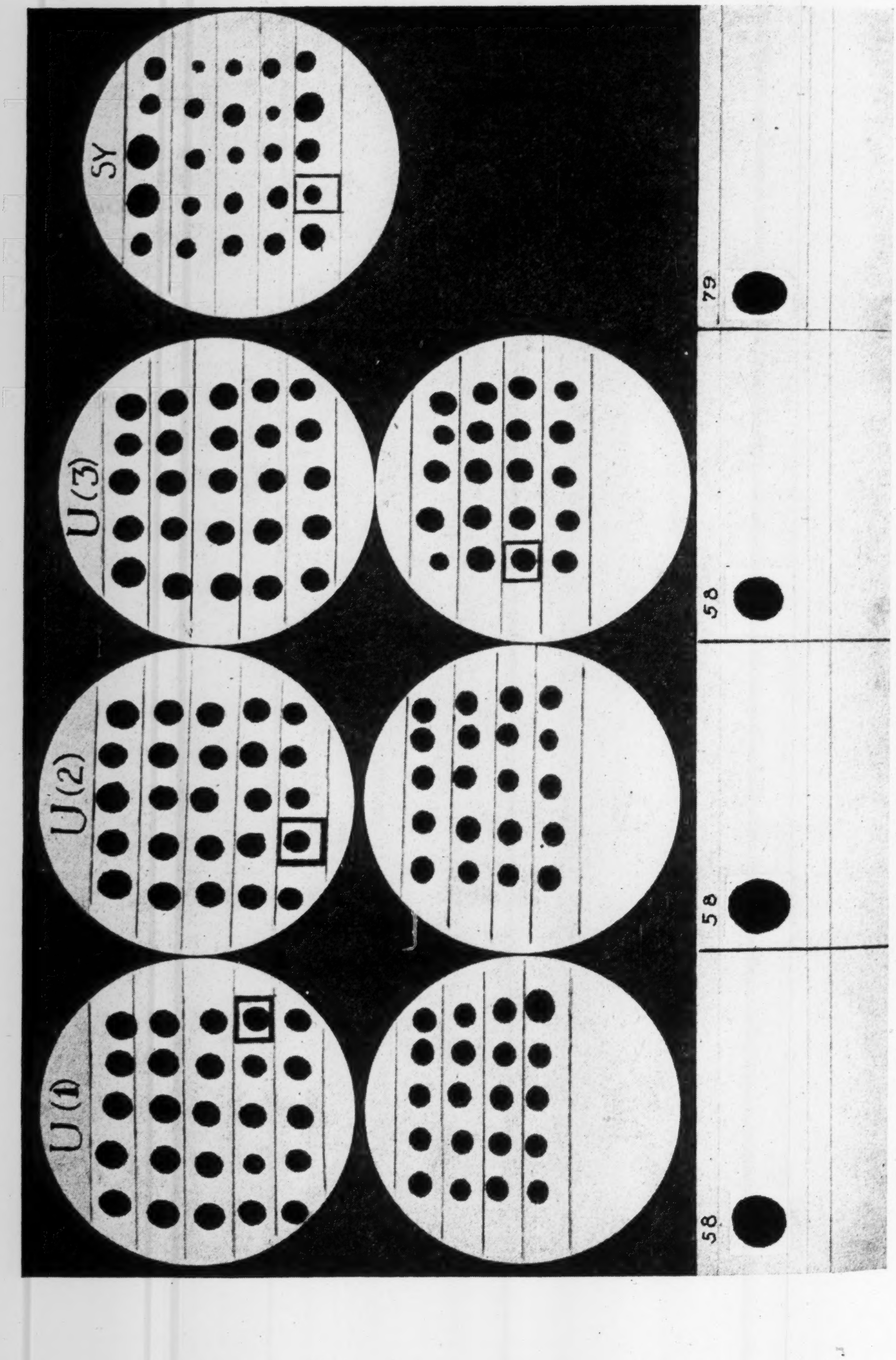
U(1)
U(2)
U(3)
SY
58
58
58
79

FIG. 3.—Quantitative Homogeneity Tests on Urbana and "Synthetic" Residuals.

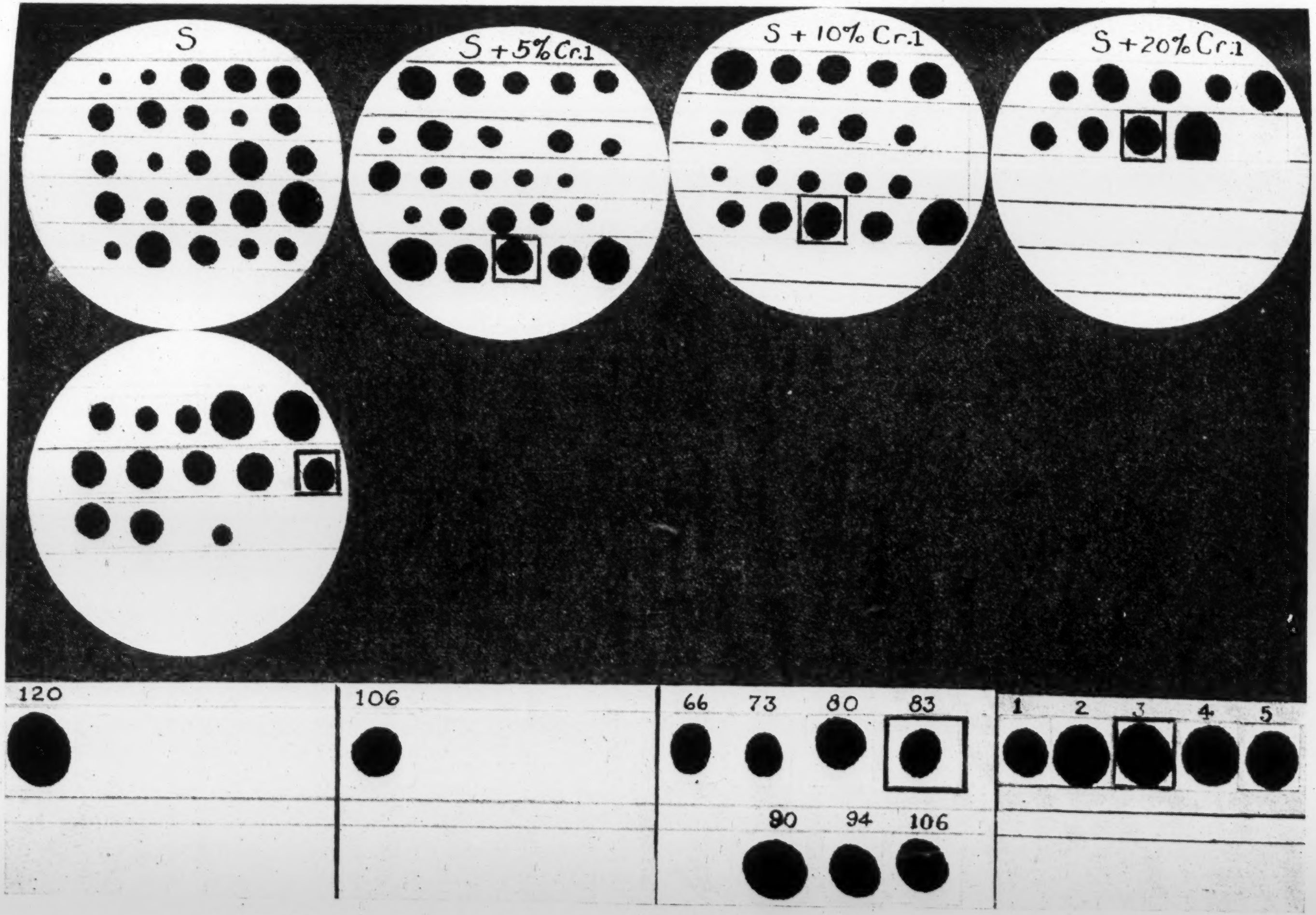
S
S + 5% Cr.1
S + 10% Cr.1
S + 20% Cr.1
120
106
66
73
80
83
90
94
106
1
2
3
4
5

FIG. 4.—Quantitative Homogeneity Tests on Blends of Smackover Residual with Cracking Coil Tar.

entitled "The Colloidal Stability of Asphalts as Indicated in Spot Tests,"[10] in which they described a very comprehensive series of spot tests they had made on different asphalts, using as the diluent both the standard naphtha and a variety of other organic solvents. While the data presented therein were well worthy of study, the work the authors had done with hexane was of particular interest. They had found that hexane yielded a positive spot with all asphalts, and had consequently determined on a number of asphalts what they had termed the "hexane-naphtha ratio"—that is the minimum percentage of hexane (in a solution of hexane and standard naphtha) required to cause a negative asphalt to yield a positive spot when the mixed solvent was substituted for the standard naphtha in the standard spot test. They had found that each negative asphalt had its characteristic hexane-naphtha ratio.

While the hexane used by Winterkorn and Eckert was by no means the pure hydrocarbon that the author had been hoping to locate, yet the sharp differentiation the hexane-naphtha ratio made between different negative asphalts appeared highly promising. Therefore, after the author had assured himself that the hexane could be obtained in commercial quantities in at least as uniform a grade as the standard naphtha he had been using satisfactorily for the past 8 yr. in the standard spot test, he proceeded to make a series of hexane-naphtha ratio determinations on a number of negative residuals, following the procedure that had been discovered by Winterkorn and Eckert. Unfortunately, one difficulty was encountered in connection with that test: the hexane was so volatile that up to one half to two thirds of it would evaporate during the cutting of the asphalt—in spite of the substitution of a 100-ml. pipette as the reflux condenser in place of the usual 8-in. length of $\frac{1}{4}$-in. tubing. It was clear that if asphalts of still higher melting point were to be tested, even higher hexane-evaporation losses, and consequently serious discrepancies in test results, might be anticipated from time to time from that cause.

After additional experimentation, however, the author developed the following method, in which hexane is still employed as the precipitating reagent; but it is added in small increments, at room temperature, and after the asphalt has already been cut in the standard naphtha as in the regular spot test. The specification for hexane, and the exact procedure for employing it, follows:

SPECIFICATION FOR HEXANE[11]

The hexane is a straight-run overhead distillate from Mid-Continent crude oil, free from cracked products of any kind, and conforming to the following specifications:

A.P.I. gravity at 60 F. (15.5 C.)	75.0 to 76.0
Distillation:	
Initial boiling point	not less than 140 F. (60.0 C.)
End point	not more than 160 F. (71.1 C.)
Aniline number[12]	138 to 145 F. (58.9 to 62.8 C.)

QUANTITATIVE METHOD FOR HOMOGENEITY-HEXANE RESISTANCE

1. Preparation of Standard Asphalt-Naphtha Mixture:

The asphalt is dissolved in the standard naphtha exactly as in the standard spot test, and set aside in the dark at approximately 77 F. (25 C). If negative at the end of 24 hr., there is measured out into a clean 25-ml. Erlenmeyer flask 3.9 g. of the asphalt-naphtha mixture (or that quantity that is calculated to contain exactly 4 ml. of the standard naphtha).

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[10] This paper has now been published as "Colloidal Stability of Asphalts—Spot Tests with Partial Solvents," in ***Industrial and Engineering Chemistry***, October, 1941, p. 1285.

[11] This product may be procured as Skellysolve "B" from the Skelly Oil Co., Skelly Bldg., Kansas City, Mo.

[12] The aniline number of the solvent is determined as described in the A.S.T.M. Standard Method of Test for Precipitation Number of Lubricating Oils (D 91 - 33), 1933 Book of A.S.T.M. Standards, Part II, p. 842.

2. Addition of Hexane Increments:

From a burette add 0.2 ml. (or an equivalent number of drops) of hexane, starting the stopwatch from zero the moment the addition of the hexane is begun. Immediately thereafter, replace the stopper in the flask, swirl the latter vigorously with a circular motion on the table top for 5 sec., and set aside in a tin or box of approximately the same diameter and height as the flask and lined with black paper to maintain the contents of the flask in subdued light. The entire operation of adding the hexane and swirling should require not more than 15 sec.

3. Spot Determination:

Four and one-half minutes later, or exactly 15 sec. before the expiration of the first 5-min. interval, remove the flask from its black-lined container, swirl vigorously on the table top for exactly 5 sec. as before, remove the stopper, insert the end of a clean glass or metal rod of not over 0.09-in. diameter (cut or ground off square) slightly beneath the surface of the liquid, withdraw it, and let a small drop fall from it upon a No. 50 Whatman filter paper. The size of the drop should be such that it will spread out to not over $\frac{5}{16}$ in. The operation of swirling and depositing a drop on the filter paper should take not over 15 sec., thus completing a full 5-min. cycle.

4. Completion of the Test:

Exactly 5 min. on the stopwatch after beginning the addition of the first increment of hexane, add a fresh increment of 0.2 ml. of hexane, then stopper, swirl, and replace the flask in the black-lined container. At 15 sec. before the expiration of the second 5-min. period, remove and swirl the flask, and withdraw a drop for the spot test exactly as before, in time for a third increment of hexane at the beginning of the third 5-min. period. Continue this procedure till a positive spot is obtained. If the positive nucleus or ring is very faint, additional spots are obtained at the end of at least two more successive 5-min. intervals, to make certain that the faint nucleus or ring first seen becomes progressively more marked in the succeeding spots. Faint nuclei or rings that may appear in sporadic spots but do not reappear in subsequent ones, or remain very faint and vaguely defined, are ignored as not authentic or significant.[9]

5. Reporting Hexane Resistance:

The Hexane Resistance—that is, the resistance of the asphalt-naphtha mixture to the flocculating force of the hexane—is considered equivalent to the total number of hexane increments required to give the first *authentic* positive spot, however faint.[9]

There is evidence that the true resistance of a standard asphalt-naphtha mixture to flocculation by hexane is much greater than merely proportional to the number of increments added. Whether it increases regularly enough to be computed logarithmically or in accordance with arithmetical or some other progression, cannot be stated till further work now under way is completed.

Hexane Resistance of Various Residuals:

The hexane resistance of typical negative-spot residuals from various crude oils, both foreign and domestic, as well as of one "synthetic" asphalt, is tabulated in the last column of Table I. Illustrations of the spot tests of a number of selected asphalts from the foregoing group are found in Figs. 1 to 4. In these illustrations the identity of the asphalt is in every case shown by the letter symbol at the top of each column, which symbol is in turn explained in the second and third columns of Table I.

The circular filter papers at the top and middle of each column show the spot tests taken during the hexane-resistance determination. It will be noted that these spots are arranged in five horizontal rows of five spots each for each filter paper, thus providing for 25 increments per paper and facilitating their count. The first authentic positive spot encountered in any hexane-resistance determination is shown enclosed in a hollow square. At times the spot enclosed in these hollow squares does not appear positive in the illustration, though in every such case a faint nucleus or ring does show on the filter paper.

The spots in the large rectangular areas at the foot of each column were obtained in the course of the determination of the negative period. The numeral over each such spot signifies the number of days elapsed from the start of the test. Not all spots obtained during the progress of the negative-period determinations are included, but only those illustrating the transition stage from negative to positive. In those cases where the transition to positive has not yet been reached, only the latest negative spot is shown.

The data in Table I and the illustrations in Figs. 1 to 4 show a marked parallelism between the negative period and the hexane resistance, particularly for similarly prepared asphalts of the same source and age; since all asphalts of low hexane resistance have a very short negative period while all of high hexane resistance have an extremely long negative period, and intermediate asphalts in the same series show intermediate values. All these things point to the two methods being both quantitative measures of essentially the same characteristic, whether the latter is called relative homogeneity or, for simplicity's sake, "negativeness" or some similar term.

The data in Table I show further that, just as in the case of the negative period, successive tests on like materials give closely concordant hexane-resistance results (compare items 9 and 10; 18 and 19; 21 and 22; 25 to 28 incl.); but that there is a wide range of difference in hexane resistance both between residuals from different crudes and residuals from the same crude manufactured at different refineries. The hexane resistance of the asphalts examined varies from 7 increments on an old Venezuelan residual of 30 penetration (item 20) to as high as 51 and 61 increments on two Kansas residuals of respectively 45 penetration and 150 penetration from one refinery (items 7 and 8).

These wide differences are no doubt due in part to each of the following causes: (*a*) the inherent characteristics of the crude oil base; (*b*) the age of the sample—that is, the period elapsed since its manufacture, great age apparently reducing the hexane resistance more rapidly than it does the length of the negative period; (*c*) the differences in refining treatment prevailing in different refineries—the more careful the treatment the higher apparently being the hexane resistance. Thus, the slightly overheated Wyoming residual (item 24), even though very soft, has a resistance of only 7 hexane increments (and a negative period of 2 to 3 days), whereas the very next sample of Wyoming residual (item 25), brought down to 208 penetration but more carefully refined, has a resistance of 14 increments (and a negative period of 65 to 72 days). Similarly the two samples of Urbana residuals (items 2 and 3), whose refining history indicates slight overheating, have a hexane resistance of respectively 20 and 22 increments; while their companion sample (item 4), prepared under more normal refining conditions, has the much higher hexane resistance of 36 increments.

The question naturally arises whether this difference in hexane resistance may not be due largely to the differences in asphaltene content of the various residuals, since in the hexane-resistance determination the asphalt frequently becomes fairly highly diluted by the end of the test (though in no case even remotely approaching the dilution required in asphaltene-content determinations). An examination of the data in

Table I, however, shows that though, for example, the Trinidad and California residuals have the lowest asphaltene content of all, yet their hexane increments average considerably less than those of the Texas, Kansas, and Arkansas residuals. Similarly, Smackover (item 1) has about the same asphaltene content as the Kansas residual (item 7); yet while the former has a fairly high hexane resistance, namely 30 or 31 increments, the Kansas sample has about double that resistance, namely 51 increments—among the highest so far encountered. Many additional similar instances of lack of correlation can be cited from Table I. Hence, it can be concluded that there is no necessary connection between the asphaltene content and the hexane resistance of a residual.

Effect on Quantitative Tests of Additions of "Positive" Materials

To determine the sensitiveness of the two quantitative tests herein described to the addition of various proportions of positive-spot materials, there were added to one of these asphalts (S, item 1), 5, 10, 20, and higher percentages of each of two cracking-coil tars, testing as follows:

Cracking-Coil Tar	Symbol	Specific Gravity at 25 C. (77 F.)	Float Test at 65 C. (150 F.), sec.	Xylene Equivalent, per cent	Insoluble in Pentane, per cent
No. 1	Cr. 1	....	15	96 to 100	27.03
No. 2	Cr. 2	1.016	15	85	8.92

The various blends were then subjected to both the negative period and the hexane-resistance determinations. The results of these tests are tabulated at the foot of Table I, and the spots, formed by the series of blends with 5 10, and 20 per cent of cracking-coil tar No. 1, are illustrated in Fig. 4.

From the data in the tabulation and the illustrations in Fig. 4, it will be noted that the effect of these added increments of the cracking-coil tars is to reduce substantially the negative period, and to decrease the number of hexane increments in almost exact arithmetical proportion with the increase of the cracking-coil tar content. As is to be expected, the cracking-coil tar No. 1, being manifestly a more highly-cracked product than No. 2, as evidenced by its higher xylene equivalent and its higher pentane insoluble, is more effective than the latter in reducing the hexane resistance and the negative period.

Practical Usefulness of Quantitative Tests for Homogeneity

From the data submitted, it appears that either of the two quantitative tests for homogeneity herein described will readily differentiate between the normally prepared residual, and a product from the same crude that has been slightly overheated or that has been adulterated with positive-spot materials, even though the degree of overheating or the amount of added positive-spot material is not of itself sufficient to make the resulting product positive to the standard spot test.

Though the foregoing reveals an important field of usefulness for these two quantitative tests, both to the producer and consumer, a prerequisite to accurate diagnosis is the possession of a recently-prepared sample of the normally-refined residual from the same crude, that may be used as a reference standard. This normal sample can always be obtained either direct from the refinery or (independently of the refinery) by a careful, small-scale reduction of the crude oil in a suitable laboratory steam or vacuum still. Once such a reference standard is available, any abnormality due to overheating or to the addition of positive-spot materials can be detected to a nicety by either of the two quantitative homogeneity tests here described. Which of these methods is the more efficient or authoritative it is impossible to state until additional work now under way is completed, though the hexane-resistance method appears for the present to be at least the more expeditious and practical of the two.

CONCLUSIONS

The reader of this paper, especially its first section, may have felt inclined to ask: If the primary function of the spot test is merely to differentiate between relatively stable and relatively unstable internal phase relationships (and not, as has been thought, to distinguish between cracked and uncracked asphalts), what practical purpose can be served by such a rather abstract and seemingly arbitrary differentiation? The answer is:

(1) Since all cracked and overheated asphalts are invariably characterized by a relatively unstable internal phase relationship and in the present market constitute the overwhelming majority of all asphalts so characterized, this instability of phase relationship is the very property we depend on in our spot test to differentiate in a practical sense between cracked and uncracked asphalts.

(2) Is there a difference in actual service performance between asphalts of relatively stable and relatively unstable internal phase relationship? This question has been studied by Governmental bureaus, technical societies, universities, state highway departments, and others. While some of the findings reported have not been as conclusive as might be desired, the general trend of the evidence so far is that there *is* such a difference in performance—in some respects favorable to the one class, in other respects to the other. The fact that a difference has frequently been detected justifies the existence of the spot test (and the measurement the latter furnishes of relative phase stabilities), if for no other reason than that it makes it possible to segregate for separate study these two differently performing major groups of asphalt.

(3) Since stability of internal phase relationship is gaged by the homogeneity of a solution of a given asphalt in naphtha, it follows that the test for stability of internal phase relationship (namely, the spot test) will automatically differentiate between asphalts which when made into cutbacks with naphtha are likely to remain smooth and homogeneous indefinitely and those which may develop sludging or precipitation in storage.

(4) As has been shown, the spot test, with its quantitative modifications, is capable of detecting and measuring even slight deviations of an asphalt from its "norm," especially when these are due to differences in severity of heating during processing, or to a blending of the two types differentiated by the test. The detection and measurement of such deviations are manifestly extremely important, not only in pure research and in conscientious refinery control, but also, and to a no lesser degree, in safeguarding the interests of the asphalt consumer. But it is now seen that these deviations are gaged directly by differences in relative stability of the internal-phase relationship. This should be a last convincing proof that the differentiation of the latter does indeed serve a useful and practical purpose.

DISCUSSION

MR. B. I. SCOGGIN[1] (*by letter*).—Since the Oliensis test was first presented to this Society in 1933,[2] there have been only two authoritative papers that contradict any of Mr. Oliensis' findings. One of these is the paper by Born[3] and the other is Bureau of Mines *Report of Investigation No. 3435*.[4]

I would hesitate to mention these papers of past years except that there is a direct reference to Born's paper on page 1110 of this present paper. Mr. Oliensis makes the following statement with regard to Born's paper:

"Several asphalts of this type, cited in a paper by Born, were in my discussion of that paper shown to be characterized by an incompatibility of the 'waxy bodies' type, vulnerable to even moderate refining temperatures."

In an earlier paper,[5] Mr. Oliensis makes mention of the so-called vulnerable materials: 75 per cent paraffin wax was mixed with 25 per cent of negative spot test asphalt and this mixture held at temperatures varying from 212 to 500 F. When this mixture was tested by the regular spot test procedure "a condition somewhat similar, in fact, to 'cracking' may be induced in it by temperatures much lower than would cause 'cracking' ordinarily." In 1937 in a written discussion of Born's paper[6] Mr. Oliensis states:

"Furthermore, since as little as 400 F., and even 212 F., may develop coking or some similar heat-breakdown. . . the presumption is that incipient cracking, or at any rate some type of heat-breakdown, took place. . ."

In Mr. Oliensis' 1936 discussion of the 75 per cent paraffin wax and the 25 per cent asphalt mixture he reports the formation of xylene insoluble bodies.

By 1937 in the quotation listed above these xylene insoluble bodies have developed into "coke or some similar heat-breakdown." We note that Mr. Oliensis always follows any reference to this "cracking" with some statement such as "or some type of heat-breakdown" and usually precedes such remarks with the statement "the presumption is." We agree with Mr. Oliensis that it is entirely a presumption to assume that heat had any *direct* relationship to the breakdown to which he so often refers.

Obviously the change is a change in intersolubilities that might logically be compared to a change that might be noted by taking a sample of the very best paint obtainable, material that is known to withstand even a most severe weathering, and diluting this paint by a ratio of from 5:1 to 20:1 with an incompatible thinner and the paint will break from its former homogeneous solutions and will no longer form the homogeneous film desired. I cite the above example as being a similar and parallel fact to the facts presented in Mr. Oliensis' paper of 1936. There are numerous similar circumstances of resinous materials breaking from a solution; in no case can such changes be attributed to temperature susceptibilities alone. The fact that a portion of paraffin wax was used in Mr. Oliensis' work is also more or less incidental, as a very similar condition can be brought about by adding regular "Oliensis naphthas" in the same proportions as the paraffin wax was added and then further diluting this mixture by making a standard spot test determination.

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[1] Manager, Research and Development, Anderson-Prichard Oil Corp., Col-Tex Refining Co., Oklahoma City, Okla.

[2] G. L. Oliensis, "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 715 (1933).

[3] Sidney Born, "The Homogeneity of West Texas Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 519 (1937).

[4] K. E. Stanfield, "Some Asphalts from Oregon Basin (Wyoming) Crude Oil," *Report of Investigation No. 3435*, U. S. Bureau of Mines.

[5] G. L. Oliensis, "A Further Study of the Heterogeneity of Asphalt—a Quantitative Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 498 (1936).

[6] Discussion by G. L. Oliensis of paper by Sidney Born, "The Homogeneity of West Texas Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 525 (1937).

I wish to emphasize the above facts concerning the reaction which Mr. Oliensis presumes to be cracking as I feel that this reference to cracking might well be compared to the definitions of cracking as outlined in the Bureau of Mines *Report No. 3435* and the evidence of cracking as cited by Born, since both of the authors in question were criticized rather extensively by Mr. Oliensis. Mr. Oliensis' definition and proof of his presumptions might be compared to defining the word "walking" as meaning "to walk." In other words, asphalts that do not react normally to the Oliensis spot test are asphalts (according to Mr. Oliensis) that are especially susceptible to even moderate refining and this susceptibility is proven by the reaction of the so-called vulnerable asphalt to the Oliensis spot test. We question this logic. Such precipitation and spotting of asphalts and other resinous materials by dilution with an incompatible naphtha is normal and its correlation with refining temperature *alone* is extremely remote. There are other facts concerning the asphalts referred to in Born's paper that we wish to include with this discussion:

First, the raw crude oil from which these asphalts were made (when it had not been subjected to any refining temperatures) is positive to the Oliensis spot test regardless of the fact that it has not been heated or altered from its natural state. We know that Mr. Oliensis does not recommend his test as a test on crude oil, but it is of importance here since it is another example of the so-called "vulnerable" materials in which temperature has very distant connection.

Second, the asphalt to which Born refers is a very stable material. It passes all types of oxidation tests such as the Shattuck oxidation test as well as certain film oxidation tests as described by Benson[7] in his very thorough work on oxidation of asphalt films. Stability to weathering and stability of inter-solubilities should not be confused.

The above facts substantiate Born's contention that asphalts that may sometimes show a definite correlation between a positive reaction to the Oliensis spot test and unstability indicated by low weather resistance could just as well have been detected by the use of a solvent of 60 aniline as compared with the standard Oliensis solvent of 138 aniline.

Furthermore, it is a fact of primary importance that the xylene insoluble materials reported by Mr. Oliensis in 1936 are not found in the asphalt mentioned in Born's paper and, in the 4 yr. that have elapsed since the presentation of this paper where Oliensis was challenged by Born to show the existence of any such xylene insoluble matter, there has been absolutely no evidence on this subject presented.

We are gratified by the fact that Mr. Oliensis in the last section of his present paper offers a practical quantitative test for homogeneity embodying the method of laboratory approach which is almost identical with that proposed in 1937 by Born plus the added hexane resistance test which is similar to the test presented by Benedict.[8]

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[7] J. R. Benson "Microscopic Reactions in Translucent Asphaltic Films," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 9, December, 1937, p. 102.

MR. JOSEPH ZAPATA[9] (*by letter*).—There is nothing I can add to Mr. Oliensis' theoretical discussions, but I have some information which I believe is pertinent to the practical application of the spot test.

The accompanying Fig. 1 represents five groups of asphalts from different courses: A from a Texas crude oil, B from a Mid-Continent crude oil, D from a Mexican crude oil, C from a partly cracked residue, and E from a highly cracked residue. A large sample of each material was separated in a pilot plant into asphaltenes by the use of hexane and resins and oily constituents by means of propane. These three components were then recombined in various proportions to make 30 different samples, many of which duplicated commercially available products. Each sample was tested by means of hexane for asphaltene content, and with fuller's earth to extract the resins. The results of these tests are shown in the accompanying Figs. 2, 3, and 4. Definite differences are indicated in the reaction of the resins towards the fuller's earth as well as striking differences in the percentages. The asphaltenes show some similarity, while some differences can be noted in the oily constituents.

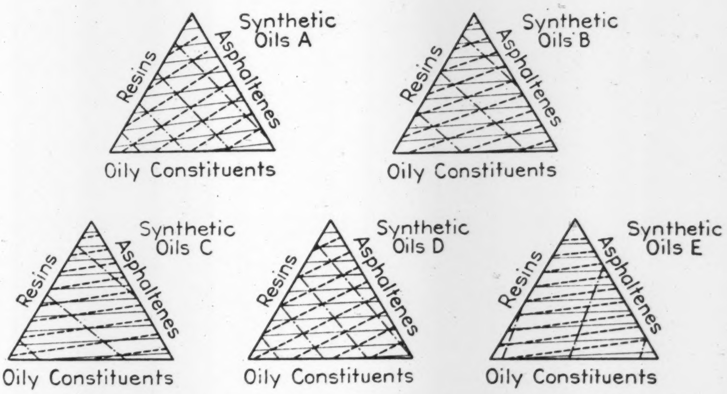

FIG. 1.—Composition of Asphalts. Laboratory analysis related to synthetic (blended) composition.

It might be said that the aforementioned results are only of academic interest; however, striking differences between positive and negative spot materials can also be observed in mixes prepared with these materials.

Using a commercially available aggregate having a gradation similar to a gradation used in road mats, mixes were made with positive and negative spot asphalts of the SC type using 11 per cent by volume of asphalt, which corresponds to percentages used in the field. By means of standard procedures, briquets were made and tested for resistance to compression and for toughness. The results of these tests are shown in the accompanying Figs. 2, 3 and 4. These results, which are typical, indicate the need for differentiating cracked from noncracked materials. This can be done readily by means of the spot test.

Mr. Oliensis has developed quantitative measurements of heterogeneity. There is a tendency to use xylene equivalent numbers as an indication of the

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[8] A. H. Benedict, "Development of the Modified Quantitative Oliensis Spot Test," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 11, January, 1940, p. 13.

[9] Senior Chemist, Laboratory of the State Highway Commission of Wisconsin, Madison, Wis.

TABLE I.—A COMPARISON OF CHARACTERISTICS OF NEGATIVE AND POSITIVE SPOT MATERIALS OF DIFFERENT XYLENE EQUIVALENT VALUES.

Furol Viscosity, 140 F., Original Samples	Spot Test, Original Sample	Xylene Equivalent, Original Sample, per cent	Xylene Equivalent of Residue. Used in making Briquettes	Compression, psi.	Resistance to Impact, number of blows
360	neg.	0	0	17.2	25
381	neg.	0	0	71.0	45
398	neg.	0	0–5	11.2	20
...	neg.	0	10	186.0	38
385	pos.	90	90	236.0	4
352	pos.	85	90	320.0	3
375	pos.	100	100	75.2	1

In order to obtain laboratory data comparable to data obtained in the field, the materials were heated for 165 min. in a container as specified in the Standard Method of Test for Residue of Specified Penetration (D 243 – 36), 1939 Book of A.S.T.M. Standards, Part II, p. 414. See Hoiberg, Hougen, and Zapata, "The Properties and Composition of Asphalts of the Slow Curving Type," *Bulletin 86*, October 1939, University of Wisconsin Engineering Experiment Station, Madison, Wis., for further details.

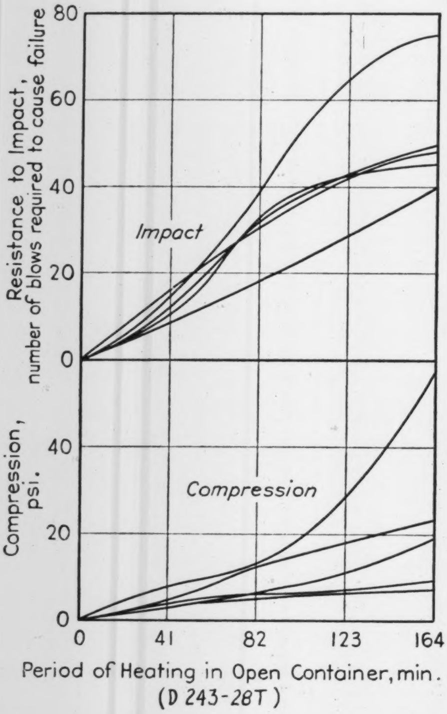

FIG. 2.—Asphalts, Type SC, Negative Spot.[10]

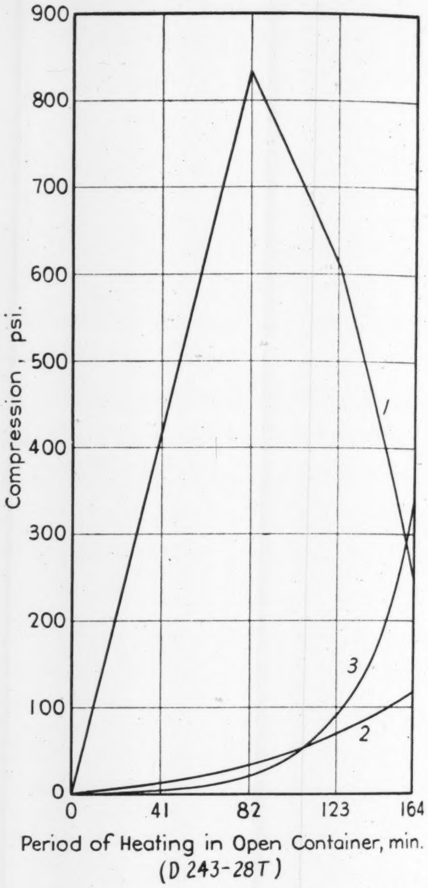

FIG. 3.—Asphalts, Type SC, Positive Spot.[10]

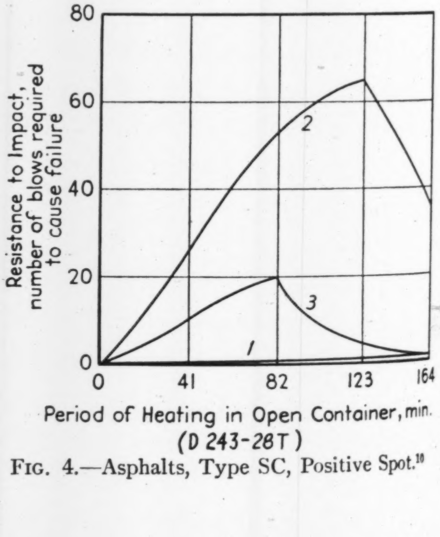

FIG. 4.—Asphalts, Type SC, Positive Spot.[10]

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[10] Data to establish the period of heating were obtained in the course of investigating changes taking place in a road mat—see *Bulletin 86*, University of Wisconsin Engineering Experiment Station.

presence of desirable qualities. The data in the accompanying Table I indicate the need for further investigations of the value of these equivalents.

Mr. Oliensis states that positive spots can be obtained with materials which have not been subjected to cracking. The accompanying Fig. 5 suggests an explanation for this observation. In the figure, triangles A, B, and D represent materials which have not been subjected to cracking; C partly cracked materials and E highly cracked residues. In triangles A and D two areas are shown, one which includes compositions which will give a positive spot test and another which comprises compositions of homogeneous materials of negative spot character. With materials of the type represented by triangle B, compositions can be made which will show a negative spot, others which will be nonhomogeneous, that is, the asphaltenes will float in the oily constituents despite efforts to disperse them. It should be noted that although we have been able to prepare, using fractions from noncracked residues, samples of compositions which show positive spots, we have not found in commercially available products compositions of similar character.

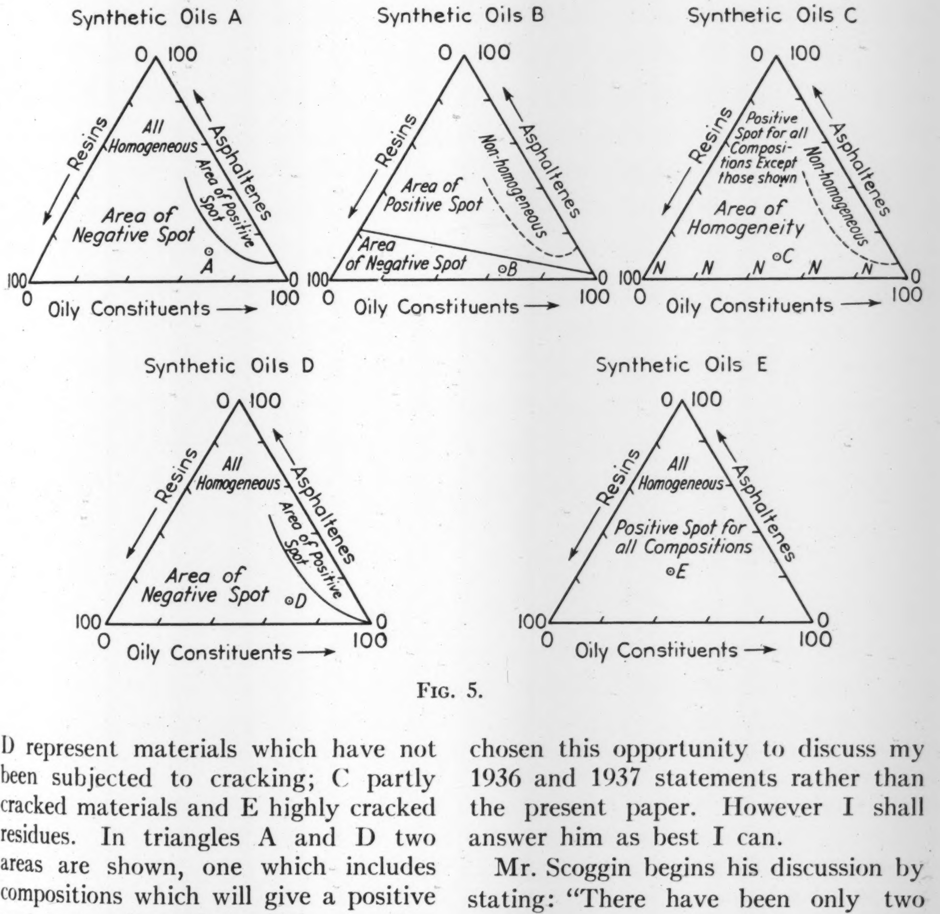
Synthetic Oils A
0
100
Resins
Asphaltenes
All Homogeneous
Area of Positive Spot
Area of Negative Spot
A
100
0
0
100
Oily Constituents →
Synthetic Oils B
Resins
Asphaltenes
Non-homogeneous
Area of Positive Spot
Area of Negative Spot
B
Oily Constituents →
Synthetic Oils C
Positive Spot for all Compositions Except those shown
Non-homogeneous
Area of Homogeneity
C
N
Oily Constituents →
Synthetic Oils D
All Homogeneous
Area of Positive Spot
Area of Negative Spot
D
Oily Constituents →
Synthetic Oils E
All Homogeneous
Positive Spot for all Compositions
E
Oily Constituents →

FIG. 5.

MR. G. L. OLIENSIS[11] (*author's closure, by letter*).—Mr. Scoggin has apparently chosen this opportunity to discuss my 1936 and 1937 statements rather than the present paper. However I shall answer him as best I can.

Mr. Scoggin begins his discussion by stating: "There have been only two authoritative papers that contradict any of Mr. Oliensis' findings"—the paper by Born and the Bureau of Mines' *Report of Investigation No. 3435*.

Mr. Scoggin is mistaken in stating that the latter report contradicts any of my findings.[12] Moreover, Mr. Scoggin is apparently unaware that two years after the appearance of *Report No. 3435* the Bureau of Mines released a final and much more comprehensive report on the same subject, in which it is stated[13]:

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[11] Chemist in Charge, Technical Dept., Barber Asphalt Corp., Madison, Ill.

> "As the positive-spot asphalts while being prepared had been subjected to higher temperatures than the negative-spot asphalts from the same crude oils, the spot tests were considered to be a measure of their degree of overheating or cracking."

Mr. Scoggin can hardly call the foregoing a contradiction of my findings and can hardly question, after this unbiased statement, the existence of a connection between cracking and overheating on the one hand and a positive-spot reaction on the other.

Nevertheless, Mr. Scoggin now reiterates the point first raised by Mr. Born in 1937 about the residuals of certain West Texas crudes. Mr. Scoggin's argument runs about as follows: That these residuals have not been cracked or overheated, and should therefore rightfully react negative to the spot test; but that since they do not react negative with the standard naphtha the fault must be with the naphtha and not with the asphalt; that hence a more compatible naphtha (by which is clearly meant a naphtha of higher solvent power than the standard) should be used so as to make these West Texas residuals pass the test, and once having passed the test they will have been demonstrated to be negative, q.e.d.! This unique line of reasoning comes from one who criticises me for lack of logic and who states that I define "walking" as meaning "to walk"!

Mr. Scoggin intimates that my past references to cracking are vague and lack definiteness. Now, none of us knows very much about the exact nature of cracking, nor is it to be overlooked that the oil refiners may not have the same concept of it as do their customers who are practical users of asphalt in paving, roofing, and waterproofing, and who may therefore have their own notions on the subject. I brought out precisely this point in my comments on T. H. Rogers' discussion of my 1936 paper,[14] wherein I said:

> "Regardless of what meaning is usually applied to the term 'cracking' in oil refinery practice, it will be generally held in the asphalt industry that when a heat reaction takes place in a pure-bitumen asphalt that develops in it substantial quantities of coked material or free carbon, totally insoluble in carbon bisulfide, this constitutes essentially a cracking reaction."

If Mr. Scoggin feels that he has a precise definition of cracking that will hold water both with the producer and consumer, let him by all means divulge it. I have no such definition to offer. My guarded references in the past to "conditions similar to cracking," my frequent linkings of "cracking" with "coking" or with some similar type of breakdown in the structure of the bitumen, were worded as they were, advisedly and after careful consideration, in order to make clear that I did not wish to tie my test down to some narrow and rigid definition of cracking; for I had already begun to visualize, even as early as 1936, the possibility of a much broader scope for the spot test. This wider scope I have for the first time attempted to describe at some length in my present paper—by pointing out that the spot test identifies, not primarily cracking, but something more fundamental, to wit: stability or instability of the internal-phase-relationship of the asphalt, and that cracking is therefore only one of several possible causes of such instability.

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[12] For a full discussion of that report see G. L. Oliensis, "Efficacy of Oliensis Spot Test on Slightly Cracked Asphalts, "*Proceedings*, Montana National Bituminous Conference, p. 193 (1939).

[13] "Report of Investigations—Asphalts from Wyoming and Other Asphalt-Bearing Crudes," *Report of Investigations 3568*, p. 27, Bureau of Mines, May, 1941.

[14] G. L. Oliensis, "A Further Study of the Heterogeneity of Asphalt—A Quantitative Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 514 (1936).

Hence I cannot quite see Mr. Scoggin's motive in reviving after so many years the old controversy about West Texas residuals—and doing so in a discussion of this present paper of all others. For even if he should now be able to accomplish the formidable task of proving that the residuals he is interested in have not been cracked, in any sense of that very broad term, he could not sidestep the more fundamental fact that since these West Texas residuals cannot stand the conditions of the standard spot test[15] that the normal straight-reduced residual can stand, the former have *ipso facto* a relatively unstable phase relationship in comparison with the latter. (Even Mr. Scoggin's reference to the precipitation resulting when a [presumably asphaltic?] paint is highly diluted with naphtha, though rather far-fetched, may be accepted as a rough illustration of the point I have just made; for such a paint could manifestly be judged to have a more "unstable internal-phase-relationship" than another similar paint which could stand the same degree of dilution without any precipitation.)

I need not enter here, of course, into a discussion either of the cause of this relative instability of phase relationship in the West Texas residuals, or of the merits of these residuals, or of the still broader question whether such relative stability or instability is desirable or undesirable in asphalts, for these matters are essentially outside the scope of the spot test itself, as the latter merely points out relative stability or instability and does not attempt to explain or evaluate it.

As to Mr. Scoggin's "challenge" of the claims I made in my 1936 paper about the development of coking or heat breakdown in asphalts containing more waxy bodies than they can tolerate, I still contend that that phenomenon does occur, has been repeated numerous times in the laboratory, and can readily be demonstrated.

One more point: I am wondering whether Mr. Scoggin means, by the last paragraph of his discussion, that my quantitative tests for homogeneity are based upon methods proposed in 1937 by Born and upon a modification of a test presented by Benedict. If so, it is sufficient to refer Mr. Scoggin to the paper in question, in which he will find it is definitely implied that my quantitative method for determining the negative period is entirely original with me, and that my quantitative method for determining hexane resistance is an outgrowth and modification of the method developed by Messrs. Winterkorn and Eckert, to whom I have given credit, and not by others.

Mr. Zapata's discussion is all of it extremely interesting, but two phases of it are particularly pertinent to this paper. One is the data in Table I and Figs. 2, 3, and 4, which show a marked difference in compression and in resistance to impact as between negative-spot and positive-spot asphalt paving mixtures. This bears out the remarks in the conclusion of my present paper, and, I might add, is entirely consistent with the increasingly large amount of similar data that is steadily accumulating in the proceedings of various societies, in the research bulletins of universities and state highway departments, and in the publications of the Public Roads Administration.

Of still more fundamental value, however, is the light cast upon the nature of "negativeness" and "positiveness" (or, stable and unstable internal-phase-relationship) by the triangular charts in Mr. Zapata's Fig. 5, indicating the degree of homogeneity of various synthetic blends of fractions derived from the asphalts from five different crude oils. They show that as one advances from the left lower apex of each triangle upwards and to the right—in other words, as one decreases the resins and increases the asphaltenes and oily constituents—the blends change from a negative to a positive type (as exemplified in triangles *A* and *D*) or from a positive type to a visibly granular texture (as exemplified in triangle *C*). Triangle *B* is especially arresting, as in it all three of the above types are progressively encountered—negative, positive, and visibly granular. No one can fail to be struck (*a*) by the parallelism between this series of progressive changes in state, and that encountered in the field of colloidal chemistry as one advances from the finest or molecular dispersions, through the intermediate or colloidal dispersions, and on to the coarsest or visible dispersions; (*b*) by the probability that the difference between negative, positive, and granular types of bitumen is essentially analogous to the difference between molecular, colloidal, and coarse dispersions—namely, one of fineness of dispersion; and (*c*) by the logical inference that the positive asphalts occupy an intermediate position between the negative and the visibly granular asphalts, analogous to that occupied by the colloids between the molecular and coarse particles.

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[15] That is, the conditions of the spot test as standardized by one technical society and being used so generally, even in patent specifications.

Nor can one avoid the inevitable conclusion that just as in colloidal chemistry it has been found that there is no sharp dividing line (either in respect to size or in respect to physicochemical characteristics) as between molecular and colloidal particles or as between colloidal and coarse particles, but that these groups merge almost imperceptibly into one another at the point of transition; so in the case of our bituminous dispersions there can be expected no sharp dividing line as between the negative and the positive, or as between the positive and the granular groups, either in respect to fineness of dispersion or in respect to stability of internal-phase-relationship, but that all these characteristics merge imperceptibly into one another during the transition from one group to the next.

If this be so, it seems futile to hope for a test capable of differentiating sharply between negative and positive, or between slightly positive and definitely positive products—unless, to take care of this gradual transition or shading from one state to the other, the test is governed by rigidly standardized conditions, as is indeed required in so many other of our tests for bituminous material, even some of the oldest ones. If these standardized conditions of the spot test are altered or tampered with, serious confusion will be bound to result; and that would be especially regrettable because it seems altogether unnecessary that these standard conditions should be altered. For the spot test, rigidly as it has been standardized, has fortunately proven flexible enough to accommodate itself to this broader understanding of the internal structure of bitumen and the gradual transitions that exist therein; and already provides in itself, with the aid of its several supplementary methods for quantitative determination, a means of simply and rapidly measuring all these gradual transitions from the negative to the positive state.

No doubt, as more and more spot test data, both qualitative and quantitative, are recorded on the various asphalts available to the industry and on the behavior of such asphalts in service, these accumulated data will ultimately be of considerable aid in determining whether positive or negative types of asphalts (and what precise degree of phase-stability or dispersion in each type) are preferable for any given service.

THE INFLUENCE OF RECOVERY TEMPERATURE ON THE DUCTILITY OF THE RECOVERED ASPHALT

By Carl Bussow[1]

Synopsis

Asphalts refined from several crudes by different refining methods are dissolved in benzol and recovered by the Bussow method, using maximum asphalt temperatures of 250 C. and 300 C. Penetration, ductility, softening point, loss on heating, and penetration after loss on heating are determined and compared with the original asphalt. The asphalts range from 15 to 100 cm. in ductility and yield conclusive data on the effect of maximum recovery temperature on the ductility of the recovered asphalt.

Steinbaugh and Brown,[2] observed that blown asphalts recovered below 200 C. had lower ductilities than the original asphalts but if the recovered asphalts were heated in a closed flask to 300 C. the ductility increased to that of the original asphalt or, in some cases, to a higher value. The asphalts used by them were oxidized in the laboratory by heating to 250 to 260 C. on the hot plate. The author believes that the oxidized components were not completely dissolved in the other components of the asphalt at 260 C. but did dissolve when the asphalt was heated to 300 C. in the recovery flask; and that the more complete solution in the latter case was the direct cause of the higher ductility obtained. The author investigated the influence of the recovery temperature on the ductility of refinery produced asphalts, both blown and steam reduced, from different crude oil stocks, as listed in Table I.

Refinery produced asphalt was used in this investigation so as to be assured of the greater uniformity of composition resulting from thorough agitation during the blowing process. It was believed that oxidizing paving asphalt coated on mineral aggregate by exposing the hot bituminous mixture to air would lead to errors inasmuch as the oxidation would occur only in a layer of minute thickness and not be general throughout the film of asphalt on the aggregate grains. The variables introduced by this condition would obscure any direct relationship between the

TABLE I.—MATERIALS.

Asphalt	Crude Stock	Refining Method	Refined at
A........	Venezuela	Steam reduced	425 C.
B........	Mexican—Panuco	Steam reduced	400 C.
C........	Colombia	Steam reduced	375 C.
D........	Texas Blend	Steam reduced	375 C.
E........	Texas Blend	Steam reduced	375 C.
F........	Mexican—Panuco	Oxidized	250 C.
G........	Colombia	Vacuum reduced, oxidized	260 C.
H........	Colombia	Vacuum reduced, oxidized	260 C.
I........	Illinois Basin	Vacuum reduced, oxidized	280 C.
J........	Texas Blend	Oxidized	260 C.

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[1] Vice-President and Chief Chemist, A. W. Dow, Inc., New York, N. Y.

[2] V. B. Steinbaugh and J. K. Brown, "A Study of Asphalt Recovery Tests and Their Value as a Criterion of Service Behavior," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. 9, p. 138 (1937).

ductility and the recovery temperature of the asphalt, the direct object of this investigation.

Procedure:

The asphalts were dissolved in an equal weight of pure benzol by boiling under a reflux condenser. Approximately 200 g. of solution were distilled in the 500-ml. flask used in the Bussow recovery method.[3, 4] As many blown asphalts are produced at about 250 C. this maximum temperature was used as well as 300 C., the usual recovery temperature recommended by the author for asphalts harder than 100 penetration.

TABLE II.—STEAM-REDUCED ASPHALTS.

	Asphalt A	Asphalt B	Asphalt C	Asphalt D	Asphalt E
Penetration at 25 C.					
Original	34	21	74	45	54
Recovered at 250 C.	35	22	76	47	56
Recovered at 300 C.	33	20	75	44	54
Ductility at 25 C., cm.					
Original	100	53	100+	50	93
Recovered at 250 C.	100	26	100+	46	98
Recovered at 300 C.	100	37	100+	48	94
Ring-and-ball softening point, deg. Cent.					
Original	60.5	69.8	48.8	58.2	56.0
Recovered at 250 C.	60.5	69.5	48.7	57.8	55.7
Recovered at 300 C.	60.7	69.9	48.9	58.0	56.0
Loss of 163 C., per cent					
Original	0.02	0.02	0.01	0.02	0.02
Recovered at 250 C.	0.21	0.20	0.06	0.20	0.16
Recovered at 300 C.	0.07	0.12	0.03	0.06	0.03
Penetration of residue at 25 C.					
Original	26	18	59	40	49
Recovered at 250 C.	27	19	59	39	48
Recovered at 300 C.	26	18	60	39	48

Effects of Recovery Temperature:

The data for the steam-reduced asphalts are given in Table II. Four out of five asphalts have the same ductility after recovery as in the original asphalt, although the maximum recovery temperatures of 250 C. and 300 C. are far below the refinery temperatures of 375 C. and 425 C. The fifth, asphalt B, refined at 400 C. behaves differently. The ductility of the asphalt recovered at 250 C. is lower than that of the asphalt recovered at 300 C., which is itself lower than the ductility of the original asphalt. An increase in temperature of recovery has brought the ductility closer to that of the original asphalt.

TABLE III.—OXIDIZED ASPHALTS.

	Asphalt F	Asphalt G	Asphalt H	Asphalt I	Asphalt J
Penetration at 25 C.					
Original	30	64	37	46	52
Recovered at 250 C.	31	64	38	47	52
Recovered at 280 C.	..	..	..	46	..
Recovered at 300 C.	29	64	37	45	50
Ductility at 25 C., cm.					
Original	29	41	85	16	16
Recovered at 250 C.	26	37	90	10	15
Recovered at 280 C.	..	..	..	16	..
Recovered at 300 C.	28	42	82	16	15
Ring-and-ball softening point, deg. Cent.					
Original	68.7	57.7	60.0	67.8	64.4
Recovered at 250 C.	68.4	57.7	59.8	67.8	64.4
Recovered at 280 C.	..	..	..	68.0	..
Recovered at 300 C.	68.5	57.9	59.9	68.0	64.5
Loss at 163 C. per cent.					
Original	0.12	0.05	0.04	0.01	0.06
Recovered at 250 C.	0.20	0.14	0.07	0.03	0.18
Recovered at 280 C.	..	..	..	0.04	..
Recovered at 300 C.	0.14	0.08	0.01	0.06	0.07
Penetration of residue at 25 C.					
Original	25	59	32	26	44
Recovered at 250 C.	26	57	32	25	43
Recovered at 280 C.	..	..	..	26	..
Recovered at 300 C.	25	58	33	25	43

In considering the other properties of the recovered asphalts, it will be noted that the maximum variations for 300 C. recovery are 1 point in penetration, 0.2 C. in softening point, 0.10 per cent in loss at 163 C., and 1 point in penetration after heating. For the asphalts recovered at 250 C. the maximum variations are 2 points in penetration, 0.4 C. in softening point, 0.18 per cent

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[3] Carl Bussow, "Notes on a Method of Recovering Bitumen from Paving Materials," *Proceedings*, Assn. Asphalt Paving Technologists, January 23, 1936, p. 160.

[4] Carl Bussow, "A Study of the Recovery of Bitumen from Soft Asphalt Cements by the Bussow Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1201 (1940).

in loss at 163 C., and 2 points in penetration. The maximum variations from the original are greater when recovered at 250 C. than at 300 C. maximum temperature but they are within the experimental error and can be considered checks of the original asphalt.

The data for the oxidized asphalts are given in Table III and show some interesting results. Asphalts F, G, H, and J, blown at 250 C., after recovery at 250 and 300 C. have the same ductility, within experimental error, as the original asphalts, but asphalt I, blown at 280 C., shows a marked loss in ductility when recovered at 250 C. while it checks the original ductility when recovered at 280 and 300 C. In no case was the ductility of the recovered asphalt raised above that of the original asphalt, except within the limits of experimental error. The other properties of the recovered blown asphalts have essentially the same maximum variations as with the steam reduced asphalts and can be considered checks of the original asphalt.

It should be noted at this point that it is unnecessary to recover the asphalt as soon as it is in solution in order to prevent hardening and loss in ductility; when the author's method of recovery is used Steinbaugh and Brown[2] have demonstrated that asphalt recovered after standing weeks in benzol solution had the same penetration and ductility as the original asphalt.

It is logical to infer that where the character of an asphalt has been altered by dispersion in a solvent in which its component parts are not all equally soluble, the less soluble will separate upon standing in solution or upon the evaporation of the solvent and will not be restored to their former condition of mutual solubility with the other components until the asphalt has been heated to the temperature at which it was originally formed or to some lower temperature that will permit the same degree of mutual solubility of the components. It is not always possible to raise the temperature of the recovered asphalt to that of its refining for, if the temperature in the recovery flask exceeds 300 C., marked cracking soon starts. That fact governed the author in adopting 300 C. as the maximum temperature in his recovery method. It was found that 300 C. was sufficient, with rare exceptions, to bring about solubility conditions that restored the original ductility and other properties of the asphalt.

Conclusion:

Asphalt can be recovered with essentially unchanged penetration and softening point at temperatures equal to, or lower than, the production temperature but the ductility is more sensitive to the temperature of recovery. Even though all the benzol has been removed, the recovered asphalt will not have its true ductility restored until it has been heated to a temperature high enough to restore the original condition of mutual solubility of its components. Usually 300 C. is sufficient for this purpose and should be used in the absence of exact knowledge of the correct refinery temperature; the ductility will not be increased in the case where the production temperature was less than 300 C.

STUDIES ON THE MEASUREMENT OF WATER VAPOR IN GASES

By F. C. Todd[1] and A. W. Gauger[2]

Synopsis

To facilitate the comparison of the measurements of the moisture content of gases by the many methods now in use, two methods are presented for the measurement of the absolute moisture content of fuel gases. These methods are applicable to both manufactured and natural gas with the impurities normally found in them. The absorption of radiation near 1.87 microns (18,700 A) by water vapor is employed for a laboratory method. The sensitivity of equipment built for the infrared spectrophotometric method is shown to be of the order of 0.25 per cent of the relative humidity at 22 C., but the effects of all the impurities have not been determined. In the portable colorimetric method for field use, fractions condensable from the gas at -70 C. are removed from an accurately measured quantity of gas and the condensate mixed with an indicator solution of cobaltous bromide in *n*-butyl alcohol. The color of the indicator solution depends on the amount of water, the amount of nonaqueous condensate and its color, the solubility of water in the condensate, and the temperature of the indicator solution. An instrument for making the colorimetric measurements is described and correction methods outlined for the above variables. The reproducibility of the measurements is discussed and shown to be good.

In the production, transportation, and distribution of fuel gases, the moisture content is of great importance in the control of corrosion and in the prevention of hydrate and ice formation in the lines and regulators. Measurements and calculations which assume Dalton's law of partial pressures for perfect gases give an accuracy sufficient for many industrial applications; however, applications do exist which demand greater precision than can be obtained by such methods. The order of the deviation from Dalton's law depends on the composition, temperature, and pressure of the gas. The quantity of water vapor may be estimated theoretically by a method devised by Keyes in which equations of state for the gas and water vapor are used and correction is made for the Poyntung effect. Using this method and available data,[3] the following gases in equilibrium with water at atmospheric pressure and 83 F. contain more water than predicted by Dalton's law by the following amounts: air, 0.87 per cent; methane, 2.3 per cent; and ethane, 2.1 per cent. Hammerschmidt[4] has shown that the error is 19 per cent for a commercial natural gas at a pressure of 800 psi.

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[1] Assistant Professor, Petroleum and Natural Gas Engineering, The Pennsylvania State College, State College, Pa.

[2] Director, Mineral Industries Research, The Pennsylvania State College, State College, Pa.

[3] Frederick G. Keyes, "Note on a Corresponding-State Equation of Practical Interest for General Physico-Chemical Computations," *Journal*, Am. Chemical Soc., Vol. 60, pp. 1761–1764 (1938).

[4] Elmer G. Hammerschmidt, "Calculation and Determination of the Moisture Content of Compressed Natural Gas," *Western Gas*, Vol. 9, pp. 9–11 (1933).

Many devices have been employed to measure the water vapor in gas. Among the methods known to have been used on natural gas lines are those that weigh the amount of water absorbed by a drying agent, that measure the temperature of the dew point, that employ the reading of wet and dry bulb thermometers (or thermocouples), and that determine the compression necessary to produce condensation. For gases that have unknown specific heats and that deviate an unknown amount from the perfect gas law, the results obtained by these methods cannot be converted by theoretical means to a common basis for comparison. The presence of heavier hydrocarbons and of the impurities found in gaseous fuels complicates experimental comparisons; for example, the methods listed cannot all be used to make comparison measurements on a gas saturated with natural gasoline.

Subcommittee VI of the Society's Committee D-3 on Gaseous Fuels, of which Mr. Gauger is chairman, considered the requirements necessary for a reference method for the comparison of the multitude of methods available and in use. In the discussion at its 1938 meeting, the subcommittee formulated these requirements substantially as follows. The absolute water content is to be measured in gaseous fuels, relatively independent of the pressure, and gaseous fuels are specified to include both natural and manufactured gas. Either gas may be partially, or completely saturated with heavy hydrocarbons and, in addition, may contain any type of impurity normally found in these gases. The composition of the gas need not be known and may vary during collection of the sample.

CHOICE OF METHODS

An extensive literature survey was made of the reported methods for the measurement of the moisture in gases in order to permit a more judicious choice of the best available methods for further investigation. Many methods have been developed for specific applications which range from the measurement of the mobility of ions as influenced by the moisture content of the gas[5] to the change in thermal conductivity of the gas as a function of the water content.[6] The requirements imposed by the statement of the problem eliminate most of these methods from consideration. The most difficult requirements are independence from the variable composition of the test gas, from the absolute pressure of the test gas, and from the possible presence of small amounts of ammonia, or hydrogen sulfide. Two methods were finally chosen for experimental investigation, the absorption of light of a certain wave length in the near infrared, and the change in color on hydration of certain halogen salts in solution in organic solvents.

The selective, quantitative absorption by water vapor of radiation at a specified wave length in the near infrared was selected as a method for a laboratory standard. The absorption by the infrared has not been used much for the quantitative measurement of very small quantities of water; however, Strong[7] recently employed the absorption in a near infrared band to measure the quantity of water in the atmosphere. The absolute sensitivity of this method in grams per unit volume is independent of the pressure. The sensitivity in terms of relative humidity increases slightly with the pressure, due to deviation from Dalton's law, provided the moisture is measured at the pressure of the transmission line from which the sample is collected. The absorption of light introduces the minimum disturbance in the gas and the selective absorption at a given wave length is perhaps the least influenced of all methods by the composition of the gas. An extensive survey of the literature revealed only one component of the fuel gases which might absorb light in the 1.87 micron (18,700 Å) absorption band of water vapor. Hydrogen cyanide is the one component which may interfere, but its absorption bands are at 1.79 and 1.9 microns and these bands are so weak that they are usually studied in liquid hydrocyanic acid.[8] Available equipment for the infrared is too expensive and too sensitive to mechanical shock to be of general commercial interest at the present time. Experimental work now in progress in several laboratories may soon alter this condition.

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[5] "The Measurement of Humidity in Closed Spaces," *Special Report No. 8*, pp. 49–52, Food Investigation, Department of Scientific and Industrial Research, England (1933).

[6] A. W. Gauger, F. C. Todd and C. C. Haworth, Jr., "On the Determination of Water Vapor in Gaseous Fuels," presented before Joint Conference of Production and Chemical Committees, Rochester, N. Y., May, 1939.

[7] John Strong, "On a New Method of Measuring the Mean Height of the Ozone," *Journal*, Franklin Inst., Vol. 231, pp. 121–156 (1941).

The change in color of salts on hydration is a reaction which is specific to the water in the gas. It will be shown that this method is relatively insensitive to the composition of the gas. The change in color of cobaltous chloride impregnated cloth or paper from blue to pink as the humidity increases has been used in weather indicators for years. Wilson[9] used this salt to indicate the presence of water in iso-amyl alcohol. Swartz[10] developed a semi-quantitative meter with a thermometer and a comparison scale printed in the colors corresponding to steps in the humidity. The Phillips Petroleum Co.[11] substituted cobaltous bromide for the chloride salt to increase the sensitivity. The indicator eventually selected for these tests is a solution of cobaltous bromide in n-butyl alcohol. To anticipate, the technique finally developed for measuring the water vapor in gaseous fuels consists of the following procedure. The water vapor and other condensables are removed by passing an accurately measured amount of the gaseous fuel through a freezing tube immersed in a bath of solid carbon dioxide in acetone, or the 24.8 per cent eutectic of hydrochloric acid. This condensate is

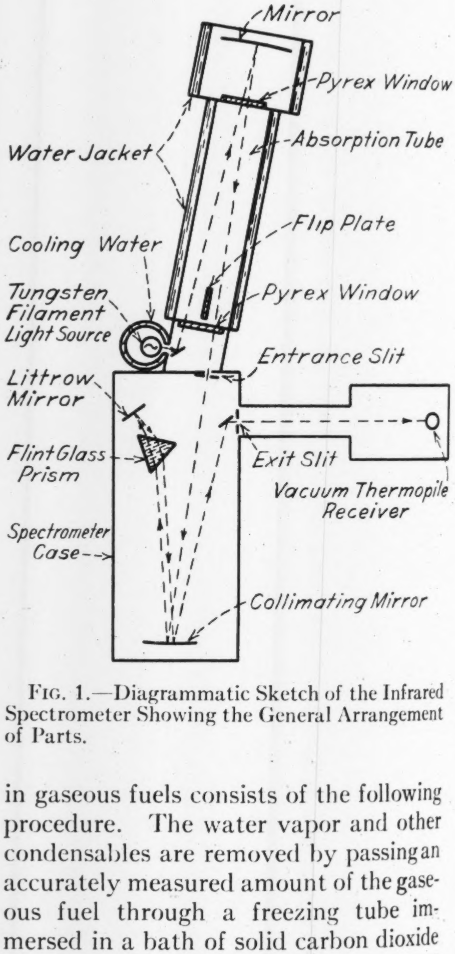

FIG. 1.—Diagrammatic Sketch of the Infrared Spectrometer Showing the General Arrangement of Parts.

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[8] F. S. Brackett and Urner Liddle, "Infrared Absorption Bands of Hydrogen Cyanide in Gas and Liquid," *Miscellaneous Collections*, Smithsonian Inst., Vol. 85, No. 5 (1931).

[9] R. E. Wilson, "Determination of Water in Iso-Amyl Alcohol," *Journal*, Am. Chemical Soc., Vol. 43, pp. 710–712 (1921).

[10] Alfred Swartz, "Color Changes in Hygroscopic Salts and Their Use for Humidity Measurements," *Messtechnik*, Vol. 9, pp. 87–92 (1933).

[11] Private communication from T. W. Legatski, Assistant Director of Research, The Phillips Petroleum Co.

dissolved in the indicator solution. Pseudotrichromatic coefficients are determined in a comparison type of colorimeter developed for this particular problem. The position of the pseudochromatic point on a calibration chart and the temperature determine both the amount of water and, roughly, the amount of condensate.

DESIGN AND SENSITIVITY OF THE INFRARED SPECTROPHOTOMETRIC METHOD

An infrared vacuum spectrometer has been built to investigate the absorption by the components of fuel gases and to determine quantitatively the molecular absorption coefficient of water in the 1.87-μ band. The spectrometer is of the Littrow type described by Brackett and McAlister[12] and shown diagrammatically in Fig. 1. The same optical arrangement is employed in an instrument built by Gaertner for the Esso laboratories.[13] In this spectrometer, the radiation traverses a lead glass prism twice to increase the dispersion. Rotation of the

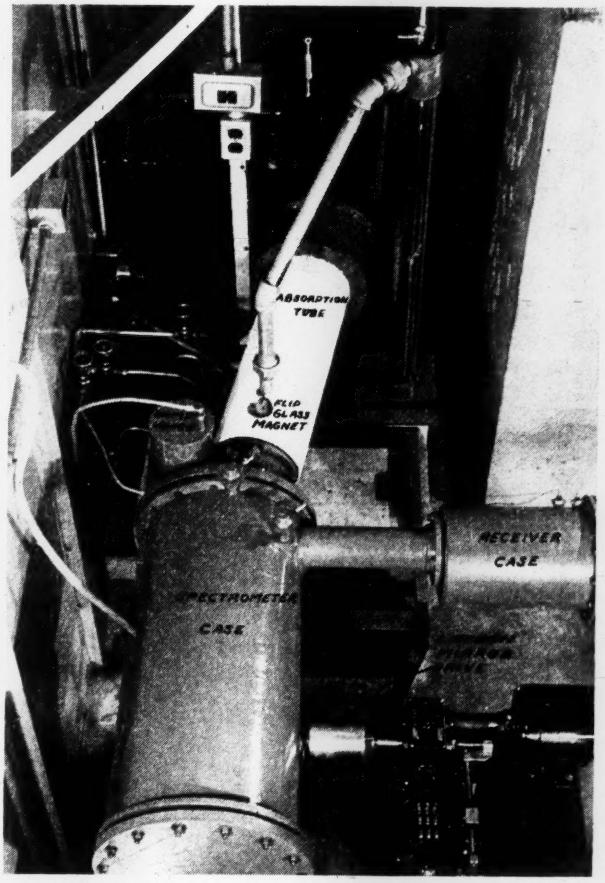

FIG. 2.—Plan View of the Spectrometer Assembly.

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[12] F. S. Brackett and E. D. McAlister, "The Automatic Recording of the Infrared at High Resolution," *Review of Scientific Instruments*, Vol. 1, pp. 181–193 (1930).

[13] E. D. McAlister, G. L. Matheson and W. J. Sweeney, "A Large Recording Spectrograph for the Infrared to 15 Microns," *Bulletin*, Am. Physical Soc., April 17, 1941, p. 14.

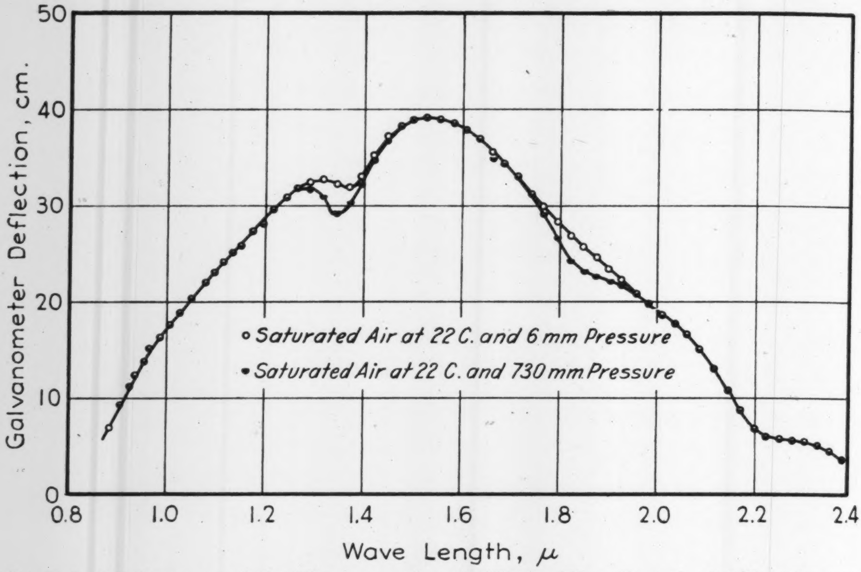

FIG. 3.—Relative Response of the Thermocouple at Different Wave Lengths for Two Pressures of Saturated Air in the Absorption Tube.

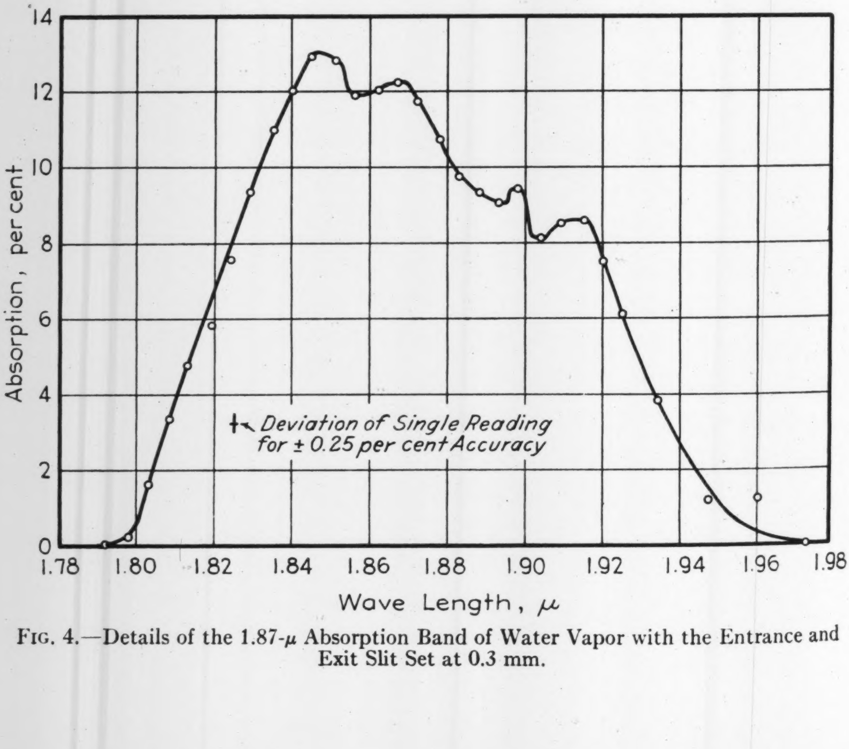

FIG. 4.—Details of the 1.87-μ Absorption Band of Water Vapor with the Entrance and Exit Slit Set at 0.3 mm.

Littrow mirror sweeps the spectrum across the exit slit. The prism is not rotated, but is set for minimum deviation at about 1.8 microns. The entrance slit is given a curvature which will result in a straight image on the exit slit at 1.8 microns. The mechanical design was radically modified so the spectrometer could be built with the limited facilities of our shop. Mr. McAlister of the Smithsonian Institution generously consented to criticize our designs. The complete spectrometer assembly, with a water-jacketed absorption tube and with the associated thermostat, is shown in the photograph, Fig. 2. The total pressure in the absorption tube is read on the manometer mounted inside the large glass tube rising from the thermostat, and jacketed in this tube by the thermostat liquid. In the measurements presented, the galvanometer deflection has been read on a scale with a 5-m. light arm to the galvanometer. A recorder, similar to the one developed by the Esso laboratories, is being assembled.

The spectrophotometric method is only semi-empirical. The concentration of the water vapor in the gas may be calculated from Lambert's law for absorption

$$I = I_0 \epsilon^{-Acd} \qquad \ldots\ldots\ldots\ldots (1)$$

where I is the transmitted intensity, I_0 is the incident intensity, d is the length of the optical absorption path, c is the concentration of the water vapor, and A is the molecular absorption coefficient for water vapor which is independent of the concentration but depends on the wave length. Lambert's law cannot be applied directly to the absorption data obtained without checking the importance of the water adsorbed on the glass windows of the absorption cell. The magnitude of this error is not yet known, so the flip glass shown in Fig. 1 is provided as a method to check its magnitude. The flip glass is of the same material as the windows of the absorption cell and it has the same polished finish on the surface. It may be inserted or removed from the light beam with a magnet. The extra glass surfaces will double the quantity of adsorbed water in the light path and provide a method to check the importance of this factor.

The investigation has only been started, but the preliminary results give the sensitivity of the method. The relative response to the radiation from a 1000-w. lamp at different wave lengths is shown in Fig. 3. The value of A, in Eq. 1, calculated from the data on this curve is 8.39. The percentage absorption by the water vapor in the 1.87-μ band, measured with slits of 0.30-mm. opening, is shown in Fig. 4. With slits of this opening, the structure in the band is beginning to appear. The sensitivity of the method depends on the smallest differences in intensities which may be measured. The straight vertical line on Fig. 4 indicates the deviations in intensity, to the scale of Fig. 4, which will correspond to ± 0.25 per cent relative humidity at 22 C., if all the intensity in Fig. 3 is employed in the measurements.

COLORIMETRIC BASIS FOR WATER MEASUREMENT

It has been noted previously that in the colorimetric method of measuring water in gaseous fuels, an accurately measured quantity of gas is passed through a freezing tube and all the components of the gaseous mixture that are condensable at the temperature of the cold bath are removed. This condensate is thoroughly mixed with an indicator solution composed of 10 g. of cobaltous bromide in a liter of *n*-butyl alcohol. Unless hydrogen sulfide or ammonia is present in considerable quantities, the factors determining the color are (1) the quantity of water in the condensate, (2) the amount of condensate other than water and its color, (3) the solubility of water in this condensate, and (4) the temperature of the solution at the time of measurement. The effect of the two impurities may be eliminated by a slight modification of the indicator solution, but no practical application has yet been found in which it was necessary.

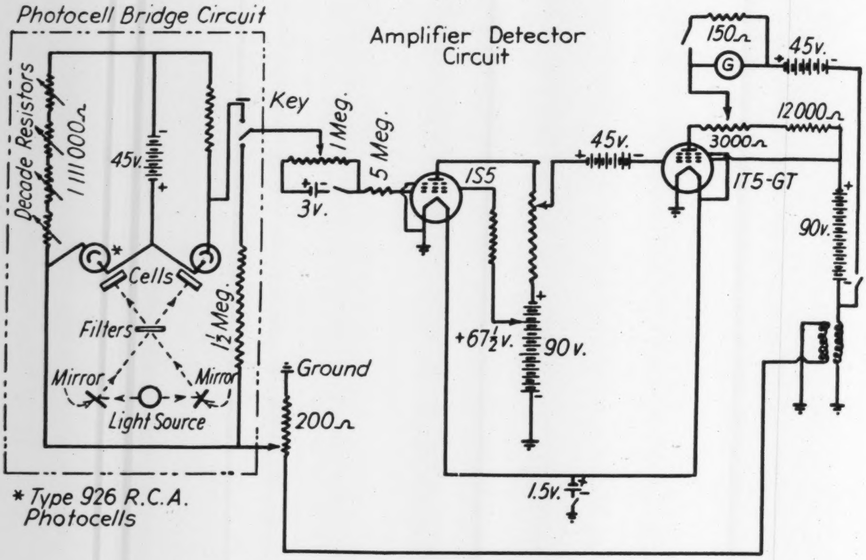
Photocell Bridge Circuit
Decade Resistors
1 111 000Ω
45v.
*
Cells
Filters
Mirror
Mirror
Light Source
1½ Meg.
Key
1 Meg.
5 Meg.
3v.
Ground
200Ω
* Type 926 R.C.A. Photocells
Amplifier Detector Circuit
1S5
45v.
+67½v.
90v.
1.5v.
150Ω
G
45v.
12000Ω
3000Ω
1T5-GT
90v.

FIG. 5.—Optical System and Electric Circuit of the Colorimeter.

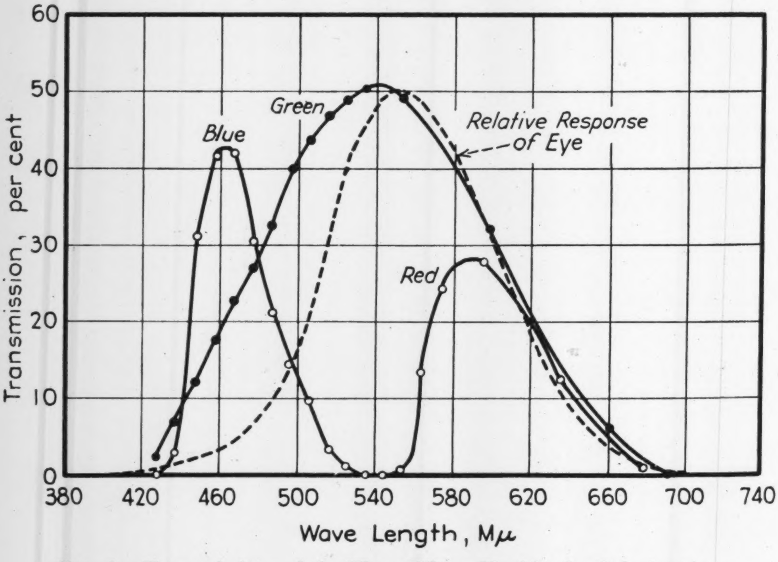

FIG. 6.—Transmissions of the Three Filters Used in the Colorimeter.

Each of these effects has been studied

by spectrophotometric curves, that is, curves which give the percentage transmission at each wave length in the visible spectrum. A set of curves which show the spectrophotometric curves for different additions of water to a solution of cobaltous bromide in *n*-butyl alcohol is given in Fig. 7. Hydrocarbon condensates increase the transmission at all wave lengths, but the ratio is not a constant. Spectrophotometric curves taken at widely different temperatures give a set of curves very similar to those in Fig. 7 for an indicator solution containing some water. Although these spectrophotometric curves completely specify the changes in color, they are cumbersome to use, difficult to interpret quantitatively, and require expensive equipment to obtain. It has been shown that spectrophotometric curves are not required for the specification of different tints, provided the same ingredients are always mixed to obtain the tint. Even more accurate measurements may be made for these colors with a filter type photometer which is cheaper and easier to operate.

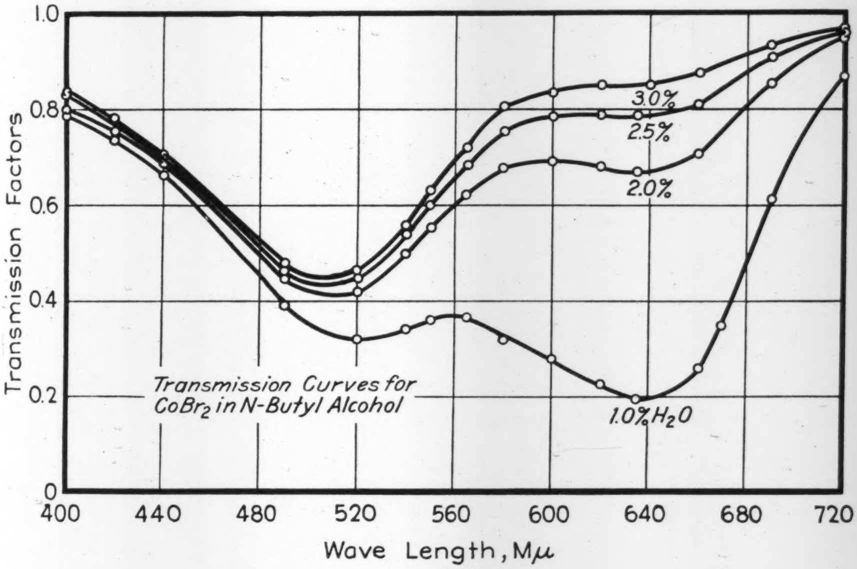

FIG. 7.—Variation in the Spectrophotometric Curves with the Addition of Water to a Cobaltous Bromide *n*-Butyl Alcohol Indicator Solution.

A comparison of different methods of treating the photometric data led to the choice of a pseudochromatic plot of the data as requiring the least computation and having the simplest corrections for the different variables. On the pseudochromatic chart, the chromaticity of each tint is calculated from readings of the percentage of light transmitted through each of three filters. The three filters were selected from a consideration of the spectrophotometric curve, Fig. 7, and the response characteristics of the photocells. The transmission characteristics of the three filters employed are given in Fig. 6. These curves were obtained with the infrared spectrometer, previously described, by operating it in the visible region with a "photronic" cell as the radiation receiver. The percentage transmissions of the colored solution, compared to clear alcohol are designated as X_0, Y_0, and Z_0, for the red, green and blue filters, respectively. The chromaticity of any tint is expressed by the pseudotrichromatic coefficients, or more properly the coordinates, which are defined by the following equations:

$$X_r = \frac{X_0}{X_0 + Y_0 + Z_0} \quad \ldots\ldots\ldots (2)$$

$$Y_g = \frac{Y_0}{X_0 + Y_0 + Z_0} \quad \ldots\ldots\ldots (3)$$

$$Z_b = \frac{Z_0}{X_0 + Y_0 + Z_0} \quad \ldots\ldots\ldots (4)$$

Since the sum of these three equations is unity, two coordinates, such as X_r and Y_g, are sufficient to specify the chromaticity completely. The location of the point obtained in this manner is determined by all the factors influencing the color. A chromatic chart of water additions to a solution of cobaltous bromide in *n*-butyl alcohol and the effect of dilution with gasoline are shown in Fig. 9. The dilutions, 5 and 10 per cent, indicate that the volume of the gasoline added was 5 or 10 per cent of the initial volume of the indicator solution. The full scale variation of the water is from zero to 1 per cent, so the hydrocarbon dilution is 5 and 10 times the volume of water required for full scale.

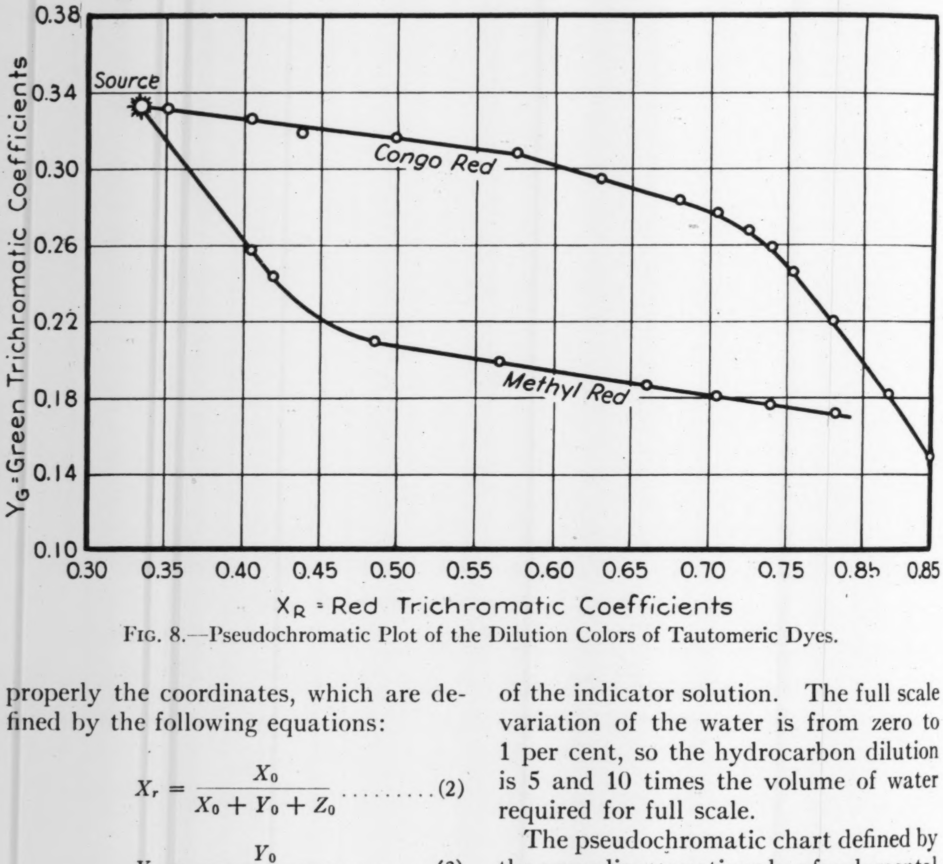

FIG. 8.—Pseudochromatic Plot of the Dilution Colors of Tautomeric Dyes.

The pseudochromatic chart defined by the preceding equations has fundamental properties particularly applicable to this problem. Unlike the true chromatic chart, the source of illumination is always represented by the coordinates 0.333, 0.333 independent of the nature of the source. "Monochromatic" colors always have coordinates far from the source, while pastel shades are near to the source. Blue colors are to the left of the source, green above, and red to the right. A basic characteristic of the true chromatic chart is the property that mixtures of two colors which do not change chemically lie along a straight line connecting the points which represent the two initial colors. This pseudochromatic chart possesses the same characteristics.

CHEMISTRY OF COLOR CHANGE

The color changes have been investigated for several salts in solution in alcohol due to the addition of water. Cobaltous iodide from our stock room was found to be decomposed, so this material is not considered sufficiently stable for general use. Nickel chloride does not have a wide variation in color. Cobaltous chloride has the same color range as the bromide, but the color variation of the bromide is about three times as sensitive to the addition of water. *N*-butyl alcohol was chosen for the solvent after extensive comparison tests with iso-amyl and iso-butyl alcohols. As an approximation, the sensitivity of the indicator solution is found to vary inversely as the solubility of water in the alcohol employed as the solvent. An indicator solution employing iso-amyl alcohol is approximately 30 per cent more sensitive than *n*-butyl alcohol, but the availability and reasonable price of the latter led to its selection.

A solution of cobaltous bromide in absolute alcohol is dark blue, but the color changes to a pale blue and, finally, to a light pink with the addition of water. A decrease in the temperature of an indicator solution containing some water will produce the same color change as the addition of water. Wilson[9] noted the color shifts with changes in temperature. He attributed the colors to the various hydrates of cobaltous chloride and suggested that the salt tends to give off water as the temperature increases, while the alcohol tends to dissolve more water due to its increased solubility. The color of the more common hydrates of cobaltous bromide are rose-red for the hexahydrate, reddish-violet for the dihydrate, and blue for the monohydrate. Several other hydrates of questionable chemical identity have been reported.[14]

The experimental results of this report are in accord with Wilson's interpretation and the known dehydration characteristics of hydrates. Colorimetric evidence has not been presented on this subject, but since the temperature correction is based on the same characteristics of the chromatic chart, this additional information will give more confidence in the method. As mentioned in a previous section, a fundamental property of the chromatic chart is that a plot of the trichromatic coefficients of mixtures of two colors which do not react chemically will lie on a straight line connecting the trichromatic coefficients of the pure colors. The chromatic plot of the change in color of the tautomeric dyes, methyl red and congo red, given in Fig. 8 shows that this characteristic is maintained for the pseudochromatic chart. The first straight line section of each dilution curve is the transition from a high concentration of one tautomeric form of the dye to a high concentration of the second form. The portions of the curves converging on the source are the dilution of this second tautomeric dye to a colorless liquid. If this curve is compared with the chromatic chart of the cobaltous bromide color changes in Fig. 11, the straight portions of the curve can be attributed to the existence of not less than three hydrates.

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[14] J. W. Mellor, "A Comprehensive Treatise on Inorganic and Theoretical Chemistry," Vol. 14, p. 712, Longmans and Co. (1937).

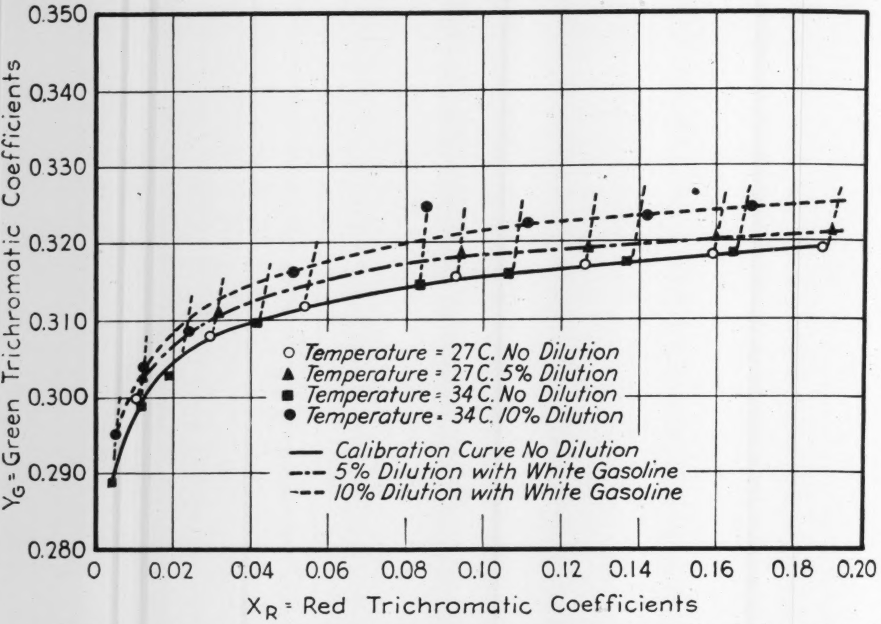

FIG. 9.—Calibration Chart Showing Effect of Temperature, and Dilution with a Clear Hydrocarbon. Filter characteristics given in Fig. 6.

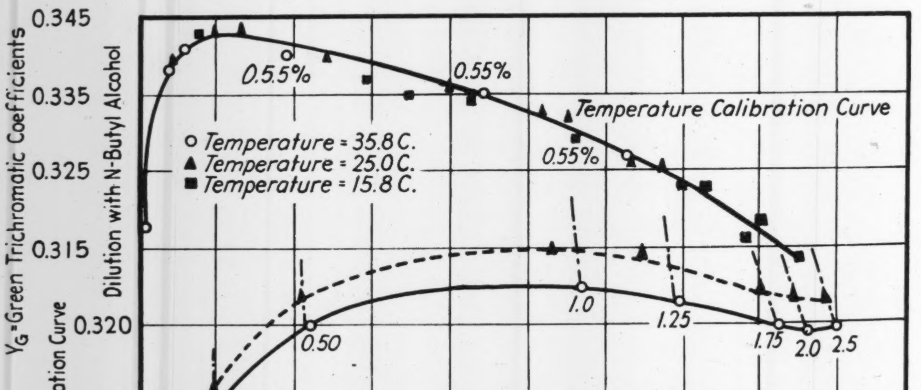

FIG. 10.—Calibration Curves with Filters Having Different Transmission Characteristics. The effect of temperature and dilution with a water-soluble dilutent.

TEMPERATURE CORRECTION WITH THE CHROMATIC CHART

The characteristics of the chromatic chart permit an easy and rapid correction for the change in color with temperature. A temperature correction is not generally feasible with the usual photometric data. If it were attempted in this case, the change in the transmission of the alcohol would be important. The average, over-all change in transmission of the combination of the optical glass cell and the clear *n*-butyl alcohol for the three filters amounts to about 1.5 per cent for a 6 C. change in temperature. The change is different for each filter. This change in absolute transmission does not introduce a similar difficulty in the chromatic plot. The chromatic curve for different amounts of water in the indicator solution of cobaltous bromide in *n*-butyl alcohol at four temperatures is shown in Fig. 11. The four points for 1 per cent water are labeled, the point farthest to the right is for the lowest temperature, 15 C. The only effect of temperature is to shift the pseudotrichromatic point for a given water content along the calibration curve. This shift along the chromatic curve is not due to a fortuitous choice of filters, but it is intrinsic in the phenomena. This is shown by the chromatic calibration charts for the different temperatures which are given in Figs. 9, 10, and 11. These calibration curves differ solely in the transmission characteristics of the color filters. The most suitable filters for this application are those which give the chart in Fig. 9. The transmission characteristics of these filters are given in Fig. 6. The effect of hydrocarbon dilution with these filters is to move the trichromatic point along

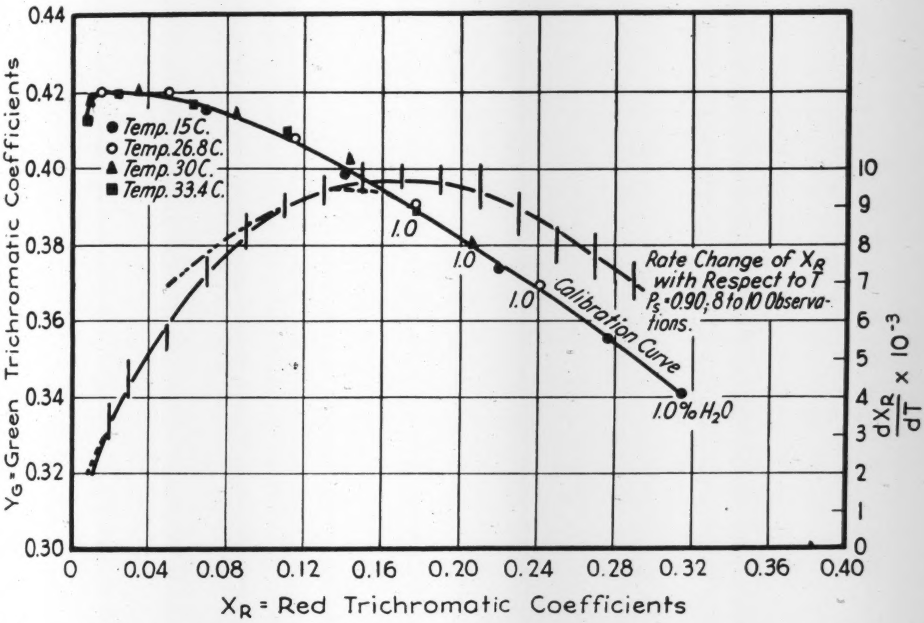

FIG. 11.—Calibration Curve for Variable Temperature, and the Variation of the Abscissa with Temperature $\left(\frac{dx_r}{dT}\right)$ as a Function of the Abscissa (x_r).

the dilution lines which make a considerable angle with the calibration curve. The dilution lines in Fig. 11 make a very small angle with the calibration curve.

Data were taken for five calibration charts similar to the upper curve in Fig. 11, but using different amounts of water as the calibration points. A plot of T *versus* x_r was drawn for each percentage of water. By the nature of the curves, from eight to ten readings of the tangent could be made for each abscissa. The lower curve is the value of the tangent, $d\,x_r/d\,T$, read at several values of the abscissa. The length of the vertical lines is calculated from the standard deviation, taking $P_s = 0.90$, 8 to 10 observations. Two equations are used to fit this curve:

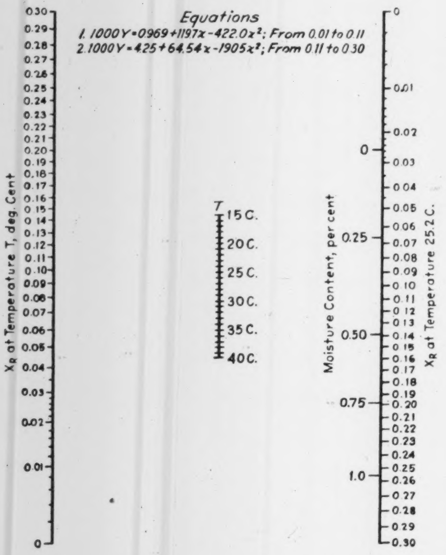

FIG. 12.—Alignment Chart for Converting Values of x_r at Temperature T C. to Moisture Content at the Standard Temperature—25.2 C.

from $x_r = 0$ to $x = 0.13$:

$$\frac{dx_r}{dT} = 0.000969 + 0.1197x - 0.4220x^2$$

from $x_r = 0.11$ to $x_r = 0.30$:

$$\frac{dx_r}{dT} = 0.00425 + 0.06454x - 0.1905x^2$$

The fit of these equations is shown by the dotted lines in Fig. 11. The fit is closer than 1 per cent of the maximum ordinate except at the lower left corner of the curve. The curve is not especially difficult to express in a single equation, but the form is more clumsy to integrate. Take the definite integral of these equations from x to $x_{25.2}$ and from T to $T = 25.2$ C., where $x_{25.2}$ is the abscissa for the calibration curve corresponding to 25.2 C. and x is the abscissa at any other temperature T. The integrals are

from $x = 0$ to $x = 0.13$:

$$0.6812 \tanh^{-1} \frac{-0.844x_{25.2} + 0.1197}{0.1263} - 0.6812 \tanh^{-1} \frac{-0.844x + 0.1197}{0.1263} = 0.04302(T - 25.2)$$

from $x = 0.13$ to $x = 0.30$:

$$\tanh^{-1} \frac{-0.381x_{25.2} + 0.0645}{0.08605} - \tanh^{-1} \frac{-0.381x + 0.0645}{0.08605} = 0.04302(T - 25.2)$$

These equations are of a simple form. Both may be represented on a single nomograph, as shown in Fig. 12. Since the values of $x_{25.2}$ are the abscissa of the calibration curve at 25.2 C., this scale of the nomograph may be calibrated in moisture content of the sample directly. This is shown on the nomograph where the moisture scale is in per cent of the initial volume of the indicator solution.

APPARATUS AND METHOD

Several methods have been investigated to bring the water in the gaseous fuel into contact with the indicator salt. The most reproducible results are obtained by removing the water vapor and other condensable matter in a freezing tube which is immersed in a bath of carbon dioxide ice and acetone. In locations in which spilled acetone may constitute a fire hazard, an even colder freezing bath, -86 C, may be obtained by mixing the 24.8 per cent eutectic of hydrochloric acid with carbon dioxide ice. A sufficient, accurately measured volume of gas at a known pressure is passed through the freezing tube to obtain approximately one-quarter gram of water. Twenty-five milliliters of indicator solution are thoroughly mixed with the condensate. The indicator solution consists of 10 g. of cobaltous bromide per liter of *n*-butyl alcohol. The chromatic coordinates of the final mixture are then determined in the colorimeter.

TABLE I.—EFFICIENCY OF FREEZING TUBES AT VARYING RATES OF FLOW.

Rate of Flow, cu. ft. per min.	Saturator Weight Loss, g.	Water Collected in Both Backing-Up Tubes, g.	Efficiency of Water Removal, per cent
0.007	2.0900	0.0000	100.00
0.017	1.4610	0.0005	99.97
0.068	0.3330	0.0010	99.70
0.12	0.4785	0.0000	100.00
0.21	0.7562	0.0000	100.00
0.22	0.4665	0.0005	99.89
0.23	0.3230	0.0000	100.00
0.35	0.8662	0.0002	99.98
0.53	0.7928	0.0002	99.97
0.55	1.4507	0.0005	99.97
0.70	0.8456	0.0005	99.94
0.72	2.5990	0.0020	99.92

Several designs of freezing tubes have been tried. The simplest design and the one which experience has demonstrated to be the most practical for use in the field consists of a U-tube of 17-mm. pyrex glass, about 14 in. high. The gas enters the U-tube and flows past a 10-in. loosely wound wire spiral of No. 18 wire. This spiral is followed by 3-in. of 5-mm. diameter, pierced glass beads; then by 2 in. of 3-mm. diameter, pierced glass beads; and finally, by 1 in. of loose cotton. The cotton removes all entrained snow and ice particles which are not caught on the other packing. This type of tube was found to be efficient in removing the moisture at flow rates to 0.70 cu. ft. per min. as shown in Table I.

All the colorimetric measurements were made on a comparison type colorimeter of special design. The comparison colorimeter employs a bridge circuit to balance the photocell currents directly. The balance of the bridge circuit is detected by a two-stage, self-contained, d-c. amplifier of nominal design. The amplifier output is fed into a bridge circuit containing a 0 to 50 microammeter. A 45-v. battery in the output bridge circuit, shown in Fig. 5, is used to permit the insertion of a more sensitive meter. This battery prevents the total plate current from the last stage of amplification from flowing through the meter. The balance of the bridge is obtained by a null method. First, the amplifier input is grounded by the key and the meter reading noted, then the amplifier input is connected across the bridge by the same key and the decade resistors adjusted to give the same meter reading. This balance is independent of the amplification and, consequently, is entirely independent of the battery voltages. The circuit shown is for the latest model colorimeter. The model on which all the readings in this report were taken differed in one essential respect. A half-silvered mirror was used to divide the light beam. With this mirror, the ratio of transmitted to reflected light depends on the wave length, the age of the mirror, and apparently, on the angle of incidence of the light.

The advantages of the first colorimeter, as established by a year of use, are listed: It is independent of fluctuations in the light source; it is independent of the amplification factor of the amplifier used as a detector except as this amplification influences the sensitivity; it employs a standard circuit which does not require delicate adjustments for stabilization; the balance is practically independent of the voltage applied to the photocells; and it is inexpensive as compared to colorimeters of comparable sensitivity. These advantages have been confirmed, but serious faults were found. The aging of the half-silvered mirror was not expected. At high humidity, leakage current made the amplifier erratic; and finally, the optical parts were not mounted with sufficient rigidity with respect to each other. The colorimeter is portable and self-contained, except for a 6-v. source of power for the light. In practice, it is portable, but a new calibration curve is required if the instrument is jarred while being transported.

TABLE II.—COLOR FILTER SPECIFICATIONS.
(Catalog Numbers for Corning Glass Color Filters)

Red Filter	Green Filter	Blue Filter
No. 978[a] No. 348	No. 978[a] No. 346	No. 038 No. 556 No. 429

[a] One third standard thickness.

The filters for the colorimeter were selected from a consideration of spectrophotometric curves of the type shown in Fig. 7; also, the spectral sensitivity of the rubidium cathode photocells and the amount of light required for an accurate balance in the bridge circuit were considered. Rough spectrophotometric curves were made of all the color changes to be expected before the first colorimeter was built or the filters selected. The specifications of the color filters are given in Table II; however, filters ordered to these specifications will not necessarily be close approximations to the filters whose transmission is given in Fig. 6. Duplicate orders of the same filters give the differences in the chromatic charts found in Figs. 9 and 11.

The exact procedure in taking a reading will be outlined. For simplicity, it will be assumed at first, that the dilutent is a colorless hydrocarbon. Sufficient, accurately measured gas is passed through the freezing tube to collect about one-quarter gram of water. This amount of water represents full scale; if less is collected, the absolute accuracy will not be as high. The condensate and packing of the U-tube is mixed thoroughly with 25 ml. of indicator solution and placed in a sealed sample bottle. The color of the solution is known to be stable for at least nine months, so the colorimetric reading may be made at any convenient time or place. The operator will have the colorimeter, a chromatic calibration chart with dilution lines similar to Fig. 9, and a nomograph similar to Fig. 12. The operator will measure the percentage transmission of the indicator solution from the sample bottle and the temperature of the solution at the time of measurement. From the three transmission measurements, he will calculate the pseudochromatic coefficients X_r and Y_g. These coefficients determine a point on the chromatic chart. If the point is above the calibration curve, the presence of a hydrocarbon dilutent is indicated. To correct for the dilution, a line is drawn from the point to intersect the zero dilution curve. This line is parallel to the adjacent dilution lines. The abscissa corresponding to this intercept and the temperature of the indicator solution are used on the nomograph, Fig. 12, to read the moisture content from the third scale. If the nonaqueous condensate is colored, or if it is soluble in water, the slope of the dilution lines must be determined by a special experiment. The dilution lines for *n*-butyl alcohol are shown in Fig. 10.

REPRODUCIBILITY

The efficiency of collection of the moisture at atmospheric pressure and at different rates of flow is shown in Table I. Manufactured gas available in the local main was passed through a freezing tube, then through a drying tube, and finally through a saturator. The wet gas was then passed through a standard freezing tube, and, finally, any moisture remaining in the gas was trapped in a drying tube and another freezing tube. The drying agent, dehydrite, employed in the check tube was the same which was used in the drying tube preceding the saturator. In all but two of the twelve test runs, the freezing tube collected 99.9 per cent of the moisture in the gas. The gas leaving the freezing tube must contain sufficient water to saturate the gas at the temperature of the freezing bath. At atmospheric pressure, the error introduced in this manner is negligible, but a correction may be required at higher pressures. If the gas is saturated at -7 C. and at 500 psi., then expanded to atmospheric pressure and passed through the freezing tube, roughly 1.9 per cent of the water will not be collected. Since the vapor pressure is known at the bath temperature, a correction may be added on the basis of Dalton's law. Even if this law is in considerable error at the low temperature of the bath, the total liquid content will be correct to one-quarter per cent, or more accurately than the over-all measurement.

The color of the indicator solution is permanent. Cobaltous bromide, reported twenty years in age, was mixed with *n*-butyl alcohol recovered by a simple batch distillation from a mixture containing crude petroleum and water. This indicator solution was allowed to stand on a shelf, tightly sealed, but exposed to light for nine months. It was then compared with a fresh indicator solution made from fresh, commercial *n*-butyl alcohol and cobaltous bromide freshly delivered from the chemical supply company. The old materials deviated less than 0.3 per cent from the calibration curve made with the fresh stock. The results were checked with a solution five months in age; this deviated only 0.2 per cent from the calibration curve. The deviations in both cases are of the order of reproducibility of the colorimetric measurements.

None of the condensates collected in field tests on the water content of the blow gas from a water gas generator has reacted with the indicator solution. Sufficient quantities of hydrogen sulfide or ammonia will react with the standard indicator to form a precipitate, but the reaction may be inhibited by dissolving dry hydrogen bromide in the indicator solution. The sensitivity to water is reduced in proportion to the amount of hydrogen bromide added, so none should be added unless necessary and then only as much as is needed.

The effect of both temperature and dilution with clear hydrocarbons is shown in Fig. 9. The slope of the dilution lines is determined from other data. The points for 10 per cent dilution at 34 C. were taken last, and the colorimeter was moved during the collection of the data so some of the spread in the data may be due to lack of rigidity in the optical system. The slope of these dilution lines is common to all hydrocarbons tried: kerosine, hexane and gasoline. If water is soluble in the condensate, the slope of the dilution lines is different, as is shown by Fig. 10 for dilution with *n*-butyl alcohol.

Color in the condensate will also change the slope of the dilution lines. For either of these types of condensate, a special calibration curve must be made. This is a simple procedure, a sample of condensate is obtained, the water removed and a calibration curve obtained by adding the dry condensate to the indicator solution. If relatively small amounts are added, the fundamental property of the chromatic chart assures that the dilution lines will be straight.

The reproducibility of the readings of the color with the colorimeter was checked by adding the same amount of water to four samples of indicator solution and measuring the chromatic coefficients of the mixtures for a total of twenty-eight readings. The calculated standard deviation of the ordinate was 0.30 per cent and of the abscissa 0.36 per cent. These readings were taken at constant temperature of the solutions. The reproducibility of the readings when the temperature is not controlled has not been determined. The nomograph does introduce some error.

The equipment has not yet been developed sufficiently for the colorimetric method to be checked for accuracy against the infrared spectrometer. It is impossible to obtain sufficient thoroughly mixed gas of constant humidity to check the over-all reproducibility. The curve in Fig. 13 shows the water content of samples of manufactured gas taken from the laboratory outlet at odd intervals during one day. The manufactured gas is transported from the water gas generator plant to the customers at a pressure of 15 to 25 psi. Since the pressure fluctuates with the load, the water content would be expected to vary. It is not known whether the average moisture content curve has any significance. The average relative humidity of the gas is about 10 per cent from these readings.

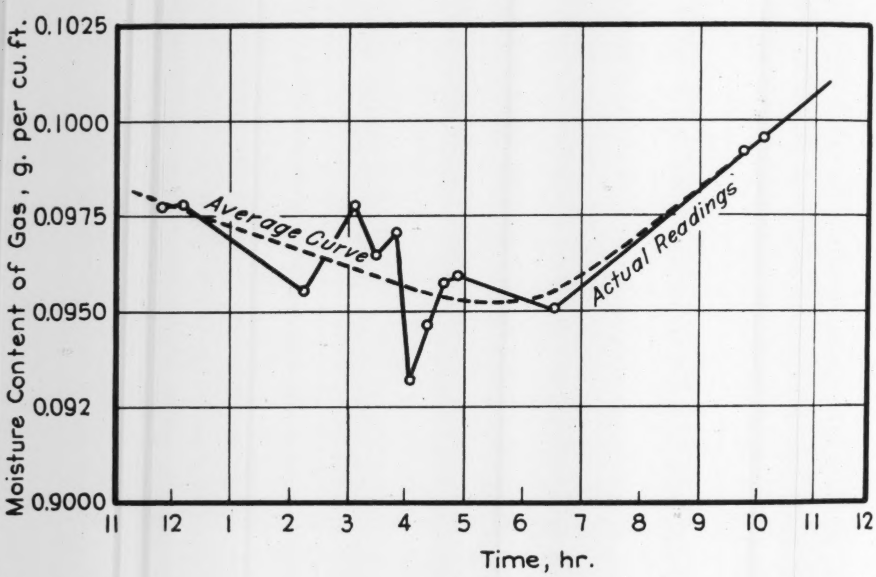

FIG. 13.—Variation in the Moisture Content of the Manufactured Gas Delivered to the College During One Day.

Acknowledgments:

The authors wish to acknowledge the grant of funds from the American Society of Testing Materials for special equipment on the spectrometer, and to the Natural Gas Supply Men's Fund for maintaining a fellowship for this work. Acknowledgment is also made to E. K. Schluntz and W. J. Wiseman, Jr., for work on the colorimetric method and to C. C. Haworth, Jr., for work on the spectrophotometric method, and particular acknowledgment is due to the mechanics in the instrument shop for their careful work in the construction of the equipment.

A STUDY OF THE GRINDABILITY OF COAL AND THE FINENESS OF PULVERIZED COAL WHEN USING THE LEA-NURSE AIR PERMEABILITY METHOD FOR EVALUATING THE SUBSIEVE FRACTIONS

By J. B. Romer[1]

Synopsis

The methods now used for determining the sieve size of pulverized coal or the particle size of finished cement are briefly discussed. The application and interpretation of the results together with their limitations show that there is a need for a better evaluation of the subsieve fractions. The use of specific surface data obtained by such methods as the Lea-Nurse air permeability apparatus is discussed, and the effect on grindability and evaluation of pulverized or finely ground products is discussed and described. It is considered that the data obtained by such methods bring the results in better alignment with the requirements of Rittinger's law.

The successful use of pulverized coal for firing metallurgical furnaces, cement kilns, boilers, etc., involves various problems that are related to the size and surface area of the coal particles. The successful transportation of pulverized coal from the pulverizer through piping, burners, and furnaces requires uniformity of size whereas ignitibility, distillation of volatile matter, and rate of combustion involve surface area.

It is well recognized that the ease with which different coals can be pulverized varies over a wide range, and in a search for a test to measure the relative grindability, a number of procedures have been developed. The interest in this subject is well evidenced by the literature and by the activities of various individuals, bureaus, and Subcommittee VII on Pulverizing Characteristics of Coal of the Society's Committee D-5 on Coal and Coke.

Grindability Procedures:

Two of the grindability test procedures are represented by the A.S.T.M. Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 - 37 T), and the Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 37 T).[2] This paper deals only with the Hardgrove-Machine method.

The grinding apparatus known as the Hardgrove grindability machine will be referred to in this paper as the grindability testing machine. This unit was the result of several years work, which had for its object the development of a suitable testing machine that would yield reproducible values. This machine and the entire method is based on Rittinger's law, which states, "The work done in pulverizing is proportional to the new surface produced."

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[1] Chief Chemist, The Babcock & Wilcox Co., Barberton, Ohio.

[2] 1939 Book of A.S.T.M. Standards, Part III, pp. 562 and 566, respectively.

Therefore, it is essential that the entire procedure should comply as nearly as possible with the fundamentals of this law.

In order to meet fully these requirements, it is necessary to measure the work done and the new surface produced. When Method D 409 was first published, it included provisions for loading the eight 1-in. grinding balls with a fixed load, namely, 64 lb. ± 0.5 lb. and required that the 50-g. sample of minus No. 16, plus No. 30 sieve coal be subjected to exactly 60 revolutions. Under these conditions, every sample receives a constant amount of work, and hence the amount of new surface produced will vary in proportion to the grindability as called for by Rittinger's law.

Since the total surface per unit weight is inversely proportional to the size of the particle, Hardgrove, in his original description of grindability **(1)**,[3] utilized sieve analysis and surface units, the latter representing the reciprocal of the average sieve opening for any given fraction.

This method of calculating surface units as $\frac{1/L_1 + L_2}{2} \times W$, where L_1 and L_2 are the nominal sieve openings and W is the weight retained on the finer sieve, was based on graphical integration where $1/L_{\text{avg.}}$ is plotted against W (total weight) and the area under the curve when multiplied by K/kp equals the total surface **(2)**. K/k is the shape factor (in general, this need not be known) and p is the specific gravity of the material. This summation is only accurate where L_1 and L_2 are known, when they are not too far apart, and when the amount of the subsieve fraction is of small magnitude.

These principles were incorporated in Method D 409 as originally written in 1935.[4] Subsequently, an empirical relationship was established between the percentage passing a 74-micron (No. 200) sieve and grindability. Therefore, this method was revised in 1937 to its present form. The empirical formula incorporated in Method D 409 is as follows:

$$\text{Hardgrove grindability index} = 13 + 6.93\,W$$

where:

W = weight of material passing the 74-micron (No. 200) sieve, determined from the weight of the original sample (50 g.) minus the weight of the material retained on the 74-micron (No. 200) sieve.

The data published by Hardgrove **(1)** showed that the amount of new surface produced decreased with the weight of sample placed in, and increased with the number of revolutions given to, the grindability testing machine; and that the grindability increased with the number of revolutions but was independent of the weight of the sample.

Present Investigation:

About a year ago, we started a review of the grindability testing machine and the test procedure for determining grindability. During the early stages of this investigation, the new surface produced was calculated from sieve analysis in accordance with the original method D 409 – 35 T.[4]

MEASUREMENT OF SUBSIEVE FRACTIONS

It has long been recognized that the knowledge of subsieve fractions was inadequate for many problems, and to overcome this difficulty there has been an ever increasing amount of work and research aimed at a suitable method for determining the particle size or surface area of subsieve fractions. Andreasen (3), Carman (4, 9), and others (8) have made large contributions to this subject. Many of these workers have been interested in other materials than coal. For example, clays, sands, ores, pigments, cements, etc., have attracted the greatest attention. Among the methods that have been considered in connection with coal, a few will be briefly discussed. They can be divided into four broad groups:

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[3] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1166.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 857; also 1935 Book of A.S.T.M. Tentative Standards, p. 629.

1. Direct count
2. Sedimentation
3. Coatings
4. Permeability

Direct Microscopic Count:

Whenever the particles that are to be counted are relatively uniform in size so that a very small sample is representative of the whole mass, and further, when the particles are of uniform geometric shape, direct count offers a method for adequately measuring either particle diameter or surface area. The greater the range of particle size and the greater deviation from any uniform geometric shape, the greater are the difficulties involved in using this method. As most samples in which we are interested show too many variations in size and shape, this method becomes altogether too tedious for commercial application.

Sedimentation:

Sedimentation is an indirect method based on Stokes' law. The technique of such tests requires constant temperature, lack of convection currents, thorough dispersion, and prolonged settling periods for the finest particles; in some methods, the fractions should be resettled in a manner analagous to chemical purification by crystallization. It has been shown by several investigators that several forms of sedimentation apparatus give results of the highest order. The Andreasen pipette is an excellent device for accurate work, but, unfortunately, the requirements of time, temperature, etc., limit its usefulness.

Coatings:

Heywood (5) and others have utilized various coatings and developed excellent methods and have provided experimental proof of incalculable value.

Permeability:

Liquids for Fluid.—Several years ago, Carman (4) proposed a method based on the Kozeny (6) equation relating the permeability of a bed of granular material to particle size. Throughout his work Carman used liquids, and wherever possible, water was selected. The results obtained are in terms of surface area of the sample, that is, square centimeters per gram of material. A mean diameter can be computed, but data on size distribution, etc., cannot be obtained. The fluid used must be one that will wet the sample without reacting with it, and the fluid must possess a molecule small enough to enter the smallest pores or capillaries between individual particles. For particles larger than about 2 microns, Carman has reported excellent data and checks.

Gases for Fluid.—Lea and Nurse (7) modified several features of the Carman apparatus. They experimented with hydrogen, helium, air, carbon dioxide, etc., and showed that the specific surface as determined was independent of the gas used. They finally adopted air as the fluid. The selection of a gas as a fluid has overcome the difficulty encountered by Carman when dealing with particles smaller than about 2 microns. Various articles in the recent literature have indicated that the Lea-Nurse air permeability apparatus will yield results comparable with the Andreasen pipette and other methods that are being used to determine specific surface.

Specific Surface

Permeability versus Turbidity:

In connection with studies of the fineness of portland cement, we have been using the Wagner turbidimeter and the test procedure as outlined in A.S.T.M. Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 – 38 T).[5] By suitable corrections, other materials than cement can be tested in this apparatus, and we have been endeavoring to evaluate subsieve fractions of pulverized coal by means of this test.

TABLE I.—DATA OBTAINED FROM LEA-NURSE APPARATUS AND WAGNER TURBIDIMETER.

Serial Number	Percentage Passing 74-micron (No. 200) Sieve	Specific Surface, sq. cm. per g.		Ratio, Lea-Nurse to Wagner
		Lea-Nurse Apparatus	Wagner Turbidimeter	
9	94.0	7945	3420	2.32
7	93.6	7800	2770	2.82
12	93.6	7535	3560	2.11
11	94.2	7510	3310	2.28
10	92.8	7295	3990	1.83
29	92.0	5285	3500	1.51
14	80.4	4920	2370	2.08
15	77.8	4850	2660	1.83
32	76.0	4585	1780	2.58
27	87.2	3665	2410	1.53

Sherman[6] of the Battelle Memorial Institute called the author's attention to the Lea-Nurse air permeability apparatus, as he considered that this piece of equipment might be of value in determining the fineness of pulverized coal and the grindability of coal by bringing the grindability method closer to the requirements of Rittinger's law.

A number of samples of pulverized coal, representing Ohio and Illinois coals, were therefore forwarded to Sherman who determined the specific surface of these samples using the Lea-Nurse air permeability apparatus and the results were compared with our earlier determinations of specific surface by the Wagner turbidimeter. The data obtained on these samples by the two methods are shown in Table I. From this it can be seen that there is no uniform relation between Lea-Nurse and Wagner data, and, as a result, there was a tendency to question the reliability of the Lea-Nurse apparatus.

TABLE II.—COMPARISON OF INDICES OBTAINED BY SPECIFIC SURFACE METHODS AND BY METHOD D 409.

	D 409 Index	Relative Indices
B-9276	100	122
B-8494	54	55
B-8495	48	47
Anthracite	29	21

The data of Table I also show that there is no accurate relationship between percentage passing a 74-micron (No. 200) sieve and specific surface. This raises the question as to our right to consider that any fundamental relation exists between such a sieve analysis and true particle size or specific surface.

Shortly after the above data had been received from Sherman, the author ground four samples of coal, namely, a soft bituminous coal, an anthracite coal, and two intermediate grindability bituminous coals in the grindability testing machine for a study at Battelle of the Lea-Nurse permeability apparatus. Sherman had expressed the thought that by enlarging the Lea-Nurse cell, the entire 50 g. could be measured before and after grinding in the grindability testing machine, thereby permitting a direct determination of the grindability. The indices normally obtained in accordance with Method D 409 were not reproduced when the product of the grindability testing machine was measured in terms of specific surface. The data are shown in Table II.

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[5] 1939 Book of A.S.T.M. Standards, Part II, p. 897.

[6] Ralph A. Sherman, private communications.

The data presented in Tables I and II indicated that the Lea-Nurse method had produced specific surface values that were in conflict with the Wagner turbidimeter data on pulverized coal and had resulted in grindability indices that were in conflict with those obtained in accordance with Method D 409. Both the Wagner method and Method D 409 are independent of one another.

In order to make a critical study of the Wagner turbidimeter, the grindability testing machine, and the Lea-Nurse air permeability apparatus and their relation to the fineness of pulverized coal and to the grindability of coal, the author had a Lea-Nurse apparatus of the same type as that at Battelle made and installed at the laboratories of the Babcock & Wilcox Co.

Wagner Turbidimeter:

The first study had to do with the Wagner turbidimeter. A mathematical study of Wagner's formula, used for calculating the specific surface of cements, indicates that particles having a specific surface greater than about 5000 sq. cm. per g. are not adequately evaluated. Therefore, whenever the sample contains appreciable quantities of material finer than this limit, the Wagner values are low. This can be written to state that cements cannot show a value in excess of about 5000 sq. cm. per g. Obviously, this limit is very questionable.

Lea-Nurse Air Permeability:

As previously stated, a duplicate of the Battelle Memorial Institute Lea-Nurse permeability apparatus was built and installed in the laboratory of the Babcock & Wilcox Co. At a later date, additional cells of larger capacity were built. Our experience indicated that a cell diameter of the order of $1\frac{1}{4}$ cm. gave the most satisfactory results, and further, that the cell length could be increased to handle larger samples. Before reproducible results were consistently obtained, considerable experimentation was required to determine the best procedure for placing and compacting the sample in the cell.

Numerous tests have shown that the specific surface could be consistently checked by different operators, if the cell containing the sample were repeatedly tapped by striking the outside of the cell with a light metal hammer, the vibrations thus set up causing the sample to pack uniformly and evenly. After this point was reached, the plunger was gradually brought in contact with the surface of the sample and the sample was further compacted by pressing the plunger downward by hand until maximum compaction was reached.

Our cell and apparatus characteristics and constants are as follows. The sample has a bed area of 1.267 sq. cm. The depth of the bed varies over a very wide range. When all of the sample passes a No. 200 sieve, the depth of the bed varies from 1.8 to 2.3 cm. For coarser fractions, and depending upon the weight involved, the depth of the bed is from 3.6 to 17.2 cm. The porosity does not vary appreciably with the particle size and usually is between the limits of 0.35 and 0.55. It is necessary to so regulate the air flow as to give suitable manometer readings and to avoid turbulent flow. This is accomplished by having two or more orifices and selecting the one most suitable for the particular sample. Our orifice or flow meter constants are 3.7×10^{-5} and

1.051×10^{-6}. The air flow may vary from as low as 6 cu cm. per min. to as high as 120 cu.cm. per min.

Without presenting too much detail, the author would like to submit the following points regarding the Lea-Nurse air permeability method.

1. A dimensional analysis of the Lea-Nurse formula

$$S_0 = \frac{14A}{W}\sqrt{\left(1 - \frac{W}{DAL}\right)^3 \times \frac{LA}{K_3} \times \frac{h_1}{h_2}}$$

shows that S_0 = square centimeters per gram.

where:

A = area of bed in square centimeters,
W = weight of particles in grams,
D = density of the particles in grams per cubic centimeter,
L = the height of the bed in centimeters,
h_1 = pressure drop across bed in centimeters of water,
h_2 = pressure drop across flow-meter in centimeters of water, and

$$K_3 = \frac{nQ}{p_2h_2}$$

where:

n = absolute viscosity of air in poises,
Q = flow rate in cubic centimeters per second,
p_2 = density of manometer liquid, and
h_2 = manometer reading in centimeters.

2. The specific surface determined is the same whether hydrogen, helium, nitrogen, air, or carbon dioxide is used as the fluid (7).

3. Any flow rate can be used that is great enough to give suitable manometer readings and not high enough to cause turbulent flow (7).

4. Excellent checks are obtained with several other methods. This feature was well brought out by various speakers at the A.S.T.M. Symposium on Particle Size Determinations (8).

5. Using sieves in the $\sqrt[4]{2}$ series, it is found that the specific surface of the various fractions is a straight-line function when plotted against average sieve opening over the range from 1190 microns to 44 microns.

6. Using carefully prepared glass beads, there is excellent agreement between specific surface by Lea-Nurse and by microscopic count (9).

7. In some instances, the specific surface of the entire product of the grindability testing machine was determined; the sample was then divided into several weighed portions, and the S_0 of each portion was then determined and the composite S_0 was calculated. In other cases, the product of the grindability testing machine was broken up by sieving and the S_0 of each fraction determined and again the composite S_0 was calculated. In all cases, good agreement was found between the different methods, thereby indicating the reliability of the S_0 measurements. For example, the S_0 of sample B-9276, after grinding in the grindability testing machine, was found to be 1485 sq. cm. per g. When the same batch was sieved into eight fractions and the S_0 of each fraction determined, then the combined S_0 was found to be 1459 sq. cm. per g.

8. The conclusion we reached with respect to this apparatus is that it is a reliable instrument for determining specific surface and can be used to determine the average particle dimension L of Rittinger's law and hence can be used for a study of the grindability testing machine and the grindability of coal.

Specific Surface and Surface Units:

For any fraction of any given material passing a given sieve and retained on the next finer sieve of the $\sqrt[4]{2}$ series, there is a direct relation between the specific surface and the new surface units calculated in accordance with Hardgrove's formula. This relation varies for different coals. By determining this relationship, the S_0 of the minus 44-micron material can be converted to surface units, or the coarser fractions can be converted to specific surface. This permits an evaluation of the product of, and the work done by, the grindability testing machine by different methods.

GRINDABILITY TESTING MACHINE

According to Rittinger's law, we can assume that within some definite limits, any grindability testing machine should produce new surface in proportion to the total work done on the coal during the test. Thus, any change from the standard procedure of loading the grinding balls with a weight of 64 lb. or from the 60 revolutions given to the top race, should be reflected proportionately in the product. To test the accuracy of this assumption, a series of tests was run wherein the above factors were varied. Raw feed to the grindability testing machine was the standard No. 16 to No. 30 sieve material, the weight of samples was varied in certain tests, and S_0 measurements were utilized in evaluating the material finer than 44 microns that was produced.

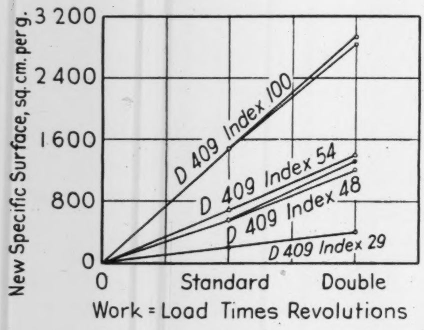

FIG. 1.—New Surface Produced *versus* Amount of Work Performed on Three Coals.

Figure 1 shows the straight-line relationship between the total new surface produced and the number of revolutions or the weight applied to the eight 1-in. balls when testing a high grindability coal and three lower grindability coals.

Figure 2 shows the relationship between specific surface produced and number of revolutions when a low grindability anthracite coal was tested. In this case, 59 g. of No. 16 to No. 30 material were placed in the machine in each test. The weight applied to the eight 1-in. balls was 64 lb. in all cases; the number of revolutions was varied.

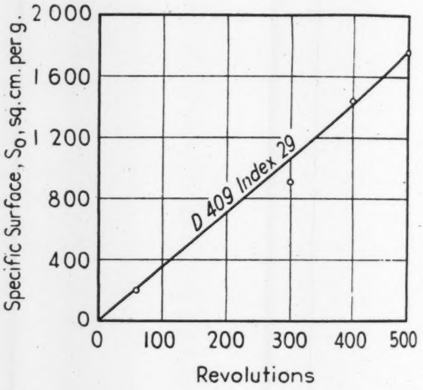

FIG. 2.—New Surface Produced *versus* Number of Revolutions Given to Grindability Testing Machine. Anthracite Coal.

The data obtained during these tests definitely show that increasing the weight or increasing the revolutions results in an increase in the weight of minus 44-micron material and in the amount of minus 74-micron material, and further, that the S_0 of the minus 44- and minus 74-micron fractions also increased. However, neither one of these increases was, in itself, proportional to the increase in the amount of work as represented by revolutions or increased weight.

The total new surface produced, nevertheless, did increase in proportion to the amount of work as represented by revolutions or weight.

In these tests the combinations of number of revolutions, weight on grinding balls, and the grindability of the coal were such as to produce a maximum of 3000 sq. cm. per g. of new surface. Within the limits used in these tests, it can be stated that if we utilize a standard coal and do the same amount of work on an unknown coal as we do on the standard, then we will obtain the same index. This will be independent of weight or revolutions.

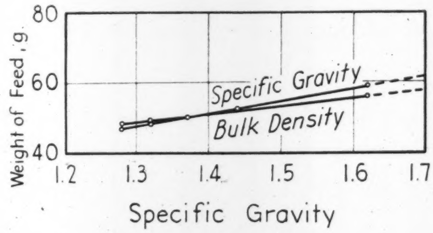

FIG. 3.—Specific Gravity *versus* Weight of Feed to the Grindability Testing Machine.

We consider that this justifies our original assumption regarding Rittinger's law and the grindability testing machine.

Specific Gravity:

The selection of 59 g. of No. 16 to No. 30 anthracite coal as the weight of material to be placed in the grindability testing machine during certain tests was based on the effect of specific gravity on the volume of the sample.

Many bituminous coals have a specific gravity which lies within the general range of 1.28 to 1.44. As the coals change in rank, the variations from this range become quite marked. Anthracite, for example, runs about 1.6 to 1.65. It is felt that the amount of material placed in the grindability testing machine should be such as to occupy the same relative volume in every test. To do this, the weight of the sample will have to be directly proportional either to the bulk density or the true density of the coal. The weight of Emore coal, which has a density of 1.37, is fixed at 50 g. Hence, for the same volume of anthracite, 56 g. are required on the bulk density basis and 59 g. are required on the true density basis. For most bituminous coals, the volume effect is too small to warrant serious comment whereas the specific gravity of anthracite is definitely beyond that of the bituminous range, and consideration of this feature is justified. Figure 3 shows the relation between weight of sample and density.

TABLE III.—DATA OBTAINED WHEN NUMBER OF REVOLUTIONS WAS VARIED SO AS TO PRODUCE CONSTANT FINENESS.

Coal	Minus 74-micron (No. 200) Sieve Material, g.	New Surface of Entire Product, sq. cm. per g.	Number of Revolutions	Index	Original D 409 Index
WEIGHT OF RAW FEED = 50 g.					
B-9276	12.8	1485	60	122	100
B-8721	12.8	1488	72	102	89
B-8723	12.8	1443	98	72	75
B-8670	12.9	1344	127	52	66
B-9240	12.9	1480	140	52	59
B-8935	12.8	1324	150	44	56
B-8993	12.7	1412	165	42	50
B-8533	12.8	1422	170	41	51
B-8936	12.8	1418	175	40	50
B-9278	13.0	1454	465	16	29
WEIGHT OF RAW FEED = 59 g.					
B-9278	2.3	200	66	16.4	29
B-9278	8.85	914	300	15.0	29
B-9278	10.75	1444	400	17.8	29
B-9278	13.0	1762	500	17.4	29

Number of Revolutions:

Having determined the relationship between number of revolutions and surface produced, a series of coals were tested to determine their grindability in terms of number of revolutions required to produce constant fineness.

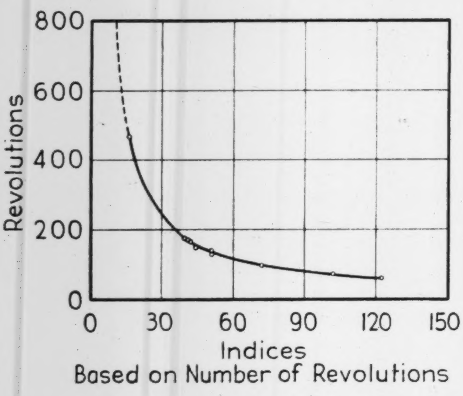

FIG. 4.—Number of Revolutions to Constant Surface *versus* New Surface Produced.

In these tests the weight of sample was, with the exception of anthracite, 50 g. and the raw feed was always No. 16 to No. 30 size.

The number of revolutions was adjusted so as to produce the same weight of minus 74-micron material in all cases. The specific surface of the entire sample after grinding was determined and the index was calculated on the relationship between new surface produced and number of revolutions in accordance with the formula:

$$\text{Index} = \frac{S_0}{670} \times \frac{60}{R} \times 55$$

where:

S_0 = square centimeters surface per gram of product,

670 = S_0 of material having a grindability index of 55,

60 = number of revolutions called for in Method D 409,

R = number of revolutions required for test, and

55 = a constant.

Table III shows the data obtained during these tests.

The indices normally obtained when these coals were tested in accordance with Method D 409 were not duplicated when tested in accordance with the procedure which has just been described.

In Fig. 4 the new indices have been plotted against the number of revolutions required to produce material having an S_0 value of 670 sq. cm. per g.

Impact Tests:

A question that is frequently raised is whether the type of grindability testing machine affects the grindability. For a study of this feature, a special impact type tester was constructed. Figure 5 is a diagrammatic sketch of this test unit which can be briefly described as follows: To a steel base-block *B*, approximately 20 in. square by 2.5 in. thick, is attached the anvil and mortar *A* and the upright rigid frame *C*. The hammer D was purposely made considerably smaller in its outside diameter than the inner diameter of the mortar. The bottom face of the hammer was parallel to the inside bottom of the mortar and the lifting rod *E* perpendicular to the same. A cover plate *F* was used to prevent the loss of fines. The rod passed through guides which

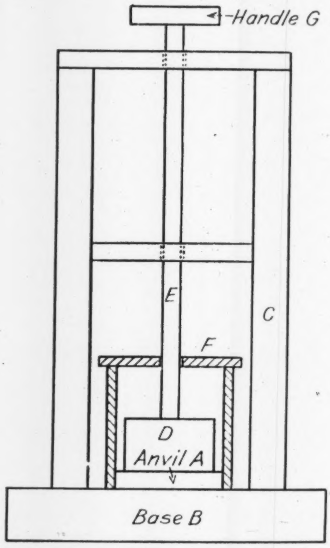

FIG. 5.—Sketch of Impact Tester.

were sufficiently free to reduce friction to the lowest possible point. The hammer weighed 22.3 lb. and was allowed to drop approximately 3 in., thus producing 5.6 ft-lb. per blow. The exact height was determined for each blow, and every ten blows the sample was loosened and mixed while in the mortar.

Following some preliminary experimentation, data were accumulated when using 100 g. of standard No. 16 to No. 30 sieve coal for the raw feed. The total work done on the coal was held between 380 and 390 ft-lb. per test. The values on five coals are given in Table IV. The new indices are calculated on the basis that 1.68 sq. cm. per g. per ft-lb. = 55.

TABLE IV.—DATA ON IMPACT TESTS.

Coal	Impact, ft-lb.	New Surface, sq. cm. per g.	New Surface, sq. cm. per g. per ft-lb.	Indices	
				Foot-pound basis.	D 409
B-9276	387.0	1440	3.72	122	100
B-8935	381.0	563	1.48	48.5	56
B-8936	381.2	543	1.42	46.5	50
B-8993	383.0	448	1.17	38.4	50
B-9278	384.3	199	0.52	17.1	29

TABLE V.—INDICES OF 72 COALS.[a]

I	II	III	IV	V	VI	VII	VIII
B-9278	29	28	22	22	15	18	17
B-8314	32	35			26		
B-8336	46	39			29		
B-8476	47	42			36		
B-9159	47.5	46	44		42		
B-8480	48	46			41		
B-8495	48	43		42	37	40	46
B-8985	49	50			46		
B-9139	49	46	50		42		
B-9141	50	53	55		49		
B-8936	50	49			45	40	46
B-8993	50	44	40	42		43	38
B-9156	50.5	50	53		46		
B-9127	51	50	51		46		
B-9140	51	57	59		56		
B-8533	51		40	40		42	
B-9160	51.5	51	45		49		
B-9154	52	56	43		56		
B-9157	52	55	54		51		
B-9095	53	56	76		53		
B-9080	53.5	49	50		43		
B-8494	54	46		49	43		48
B-9088	54	55	71		51		
B-9082	54.5	57	53		53		
B-9149	55.5	60	57		57		
B-9075	56.5	59	60		55		
B-8518	57	59			53		
B-8935	57	62	65		62	44	49
B-8618	58	61	62	62			
B-9161	58.5	64	59		62		
B-8620	59			54			
B-8521	59	56			53		
B-8595	59	55			53		
B-8499	59	59			57		
B-8453	59	59			57		
B-8528	59	49			56		
B-8621	59			60			
B-9240	59					53	
C-1728	60					50	
B-8517	60	57		51	59		
B-8470	60	64			59		
B-8642	60	60			57		
B-8535	60			55			
B-9102	61.5	73	88		71		
B-8671	62			55			
B-9145	63.5	77	85		78		
B-8631	64	66			67		
B-9072	65.5	62	65		58		
B-8670	66			58		52	
B-8619	67	63		78	66		
B-9137	68	73	74		70		
B-8674	69			74	73		
B-8482	70	72		72	72		
B-8274	73	72		78			
B-9158	75	78	81		82		
B-8723	75					73	
B-9084	77	79	77		74		
B-8445	79	81		81	81		
B-8443	80	76			83		
B-9100	80.5	85	87		85		
B-9099	81.5	78	98		90		
B-9085	82	88	84		88		
B-8721	83	100	105		88	102	
B-8323	84	93			93		
B-8271	89	88	101	101		102	
B-9134	93	105	105		115		
B-8411	94	107			118		
B-9276	100	122	122	122	122	122	122
B-8635	101	126			148		
B-8525	106	127			149		
B-9061	109	123	124		137		
B-8337	112	109			132		

[a] *Column I.*—Identification serial of coal.

Column II.—D 409 indices.

Column III.—Indices obtained by subjecting the product of the grindability testing machine to sieve analysis in accordance with the original Method D 409 – 35 T. The equivalent surface area of the plus 44-micron material was calculated in accordance with reciprocal surface units as originally described by Hardgrove (**1**); for the minus 44-micron fraction, the reciprocal surface units were calculated from the S_0 of this fraction.

19,700 total new surface units = 55

Column IV.—The product of the grindability testing machine was broken up either into several aliquots or else into several sieve fractions and the specific surface of all aliquots and all sieve fractions was determined and then the specific surface of the entire product was calculated.

$S_0 = 670$ sq. cm. per g. = 55

Column V.—The surface area of the entire 50 g. of grindability testing machine product was determined in a single large cell in the Lea-Nurse apparatus.

$S_0 = 670$ sq. cm. per g. = 55

Column VI.—Index based on the total surface of the material passing through the 44-micron (No. 325) sieve. This was calculated by determining the specific surface of this fraction and multiplying it by the grams passing the 44-micron sieve.

$S_0 \times W = 48{,}000$ sq. cm. = 55

Column VII.—Index based on the relationship between the specific surface of the entire product and the number of revolutions required to produce a fixed amount of surface.

60 revolutions at 670 sq. cm. per g. = 55

Column VIII.—Index based on impact tests.

1.68 sq. cm. per g. per ft-lb. = 55

Indices:

It has been shown that the normal Method D 409 indices were not reproduced when surface area, where S_0 equals square centimeters per gram, was used for evaluating the product of the impact tester or the grindability testing machine.

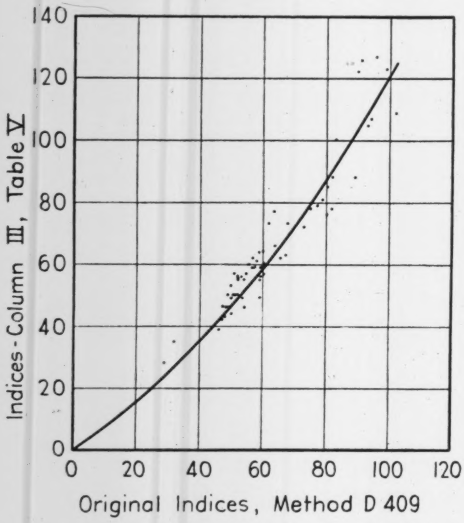

FIG. 6.—Relation Between Method D 409 Indices and Those Obtained by Adjusting New Surface Units of Minus 44-Micron Material.

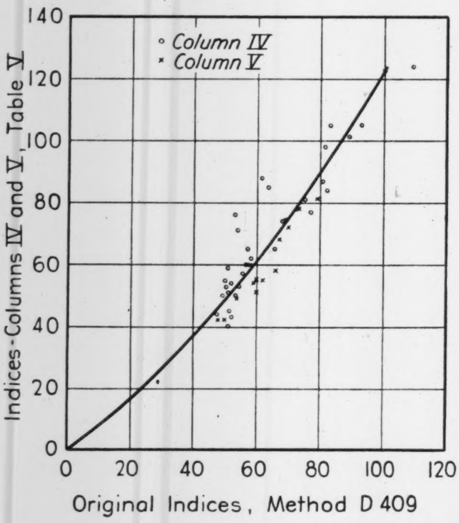

FIG. 7.—Relation Between Method D 409 Indices and Those Obtained by Determining Specific Surface of the Product of the Grindability Testing Machine.

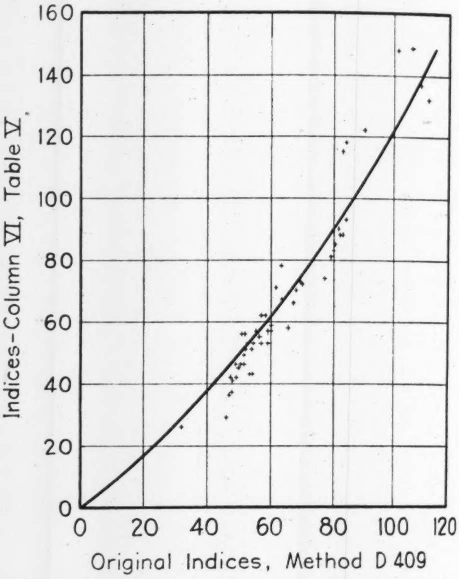

FIG. 8.—Relation Between Method D 409 Indices and Those Obtained by Calculating the Total Surface of the Minus 44-Micron Material.

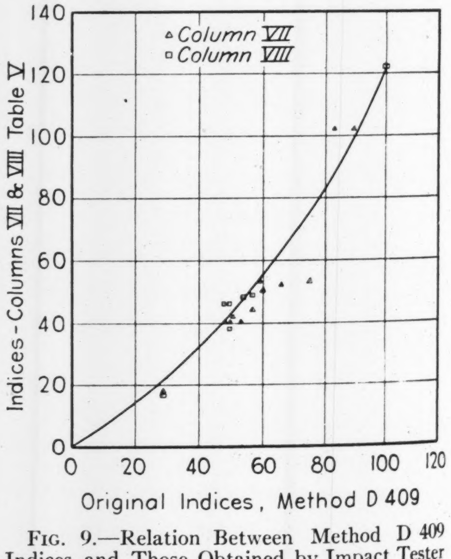

FIG. 9.—Relation Between Method D 409 Indices and Those Obtained by Impact Tester and Number of Revolutions Required to Produce Constant Surface.

Earlier in this paper, we showed that the data obtained with the Lea-Nurse air permeability apparatus produced results that were not in agreement with either the Wagner turbidimeter data or Method D 409 indices. The various tests which have been so far reported, while not agreeing with the Method D 409 indices, are in other respects in good agreement. While carrying on these tests, many samples of coal submitted to us for routine determinations of grindability were so handled that we were able to calculate the data according to several formulas, and in this way we obtained considerable inter-relation between specific surface, weight through various sieves, and grindability indices.

TABLE VI.—NUMERICAL VALUE OF PRINCIPAL COORDINATES OF FIGS. 6, 7, 8, AND 9.

D 409 Indices	Column III, Fig. 6	Columns IV and V, Fig. 7	Column VI, Fig. 8	Columns VII and VIII, Fig. 9
100.....	120	122	122	122
80.....	88	89	89	83
60.....	58	61	61	55
55.....	53	55	55	49
40.....	35	37	37	36
20.....	15	17	16	14
0.....	0	0	0	0

TABLE VII.—RELATION BETWEEN ORIGINAL METHOD D 409 INDICES AND EQUIVALENT SPECIFIC SURFACE, SQ. CM. PER G.

Original Method D 409 Indices	Average of Columns IV and V, Fig. 7	Equivalent S_0, sq. cm. per g.
100......	122	1485
80......	89	1085
60......	61	745
55......	55	670
40......	37	450
20......	17	195
0......	0	0

Table V gives all of the data we have collected regarding the indices of 72 coals when determined in various manners.

The data listed in Table V have been plotted and reproduced as Figs. 6 to 9, inclusive. These plots show good agreement and permit drawing smooth curves. Table VI lists the main points of these curves.

The final curve for interrelating specific surface indices and Method D 409 indices is shown in Fig. 10 and the main points of this curve are given in Table VII.

As a result of adding specific surface principles to the determination of grindability, the indices of softer coals rise, whereas coals having indices, according to the Method D 409 scale, of 65 or less show little change. In fact, it would appear that the present indices could be retained for the majority of coals used commercially, as they fall in the range of 45 to 65.

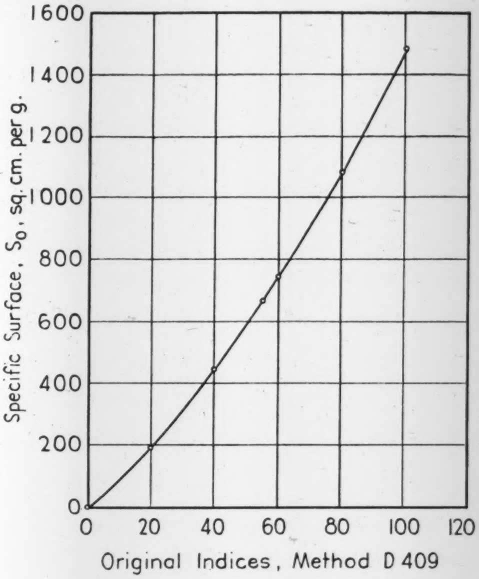

FIG. 10.—Interrelation Between Method D 409 Indices and Average of Those of Fig. 7.

SUMMARY

After reviewing the methods for determining particle size or surface area of subsieve fractions, the Lea-Nurse air permeability apparatus was selected for a study of these fractions as well as for a study of the grindability testing machine and the grindability of coal.

The test results can be summarized as follows:

1. The grindability testing machine has been shown to operate in accordance with Rittinger's law.

2. The new surface produced in the grindability testing machine is proportional to the amount of work.

3. Under conditions of constant work, the grindability is proportional to the new surface produced.

4. The indices, as a result of the

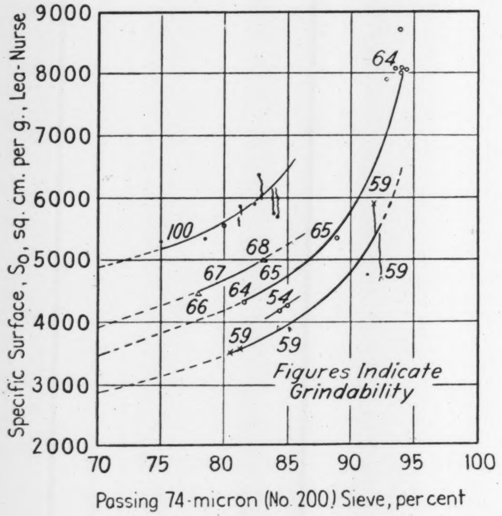

FIG. 11.—Relation Between Specific Surface and Percentage Passing 74-micron (No. 200) Sieve; 100 Series Pulverizer.

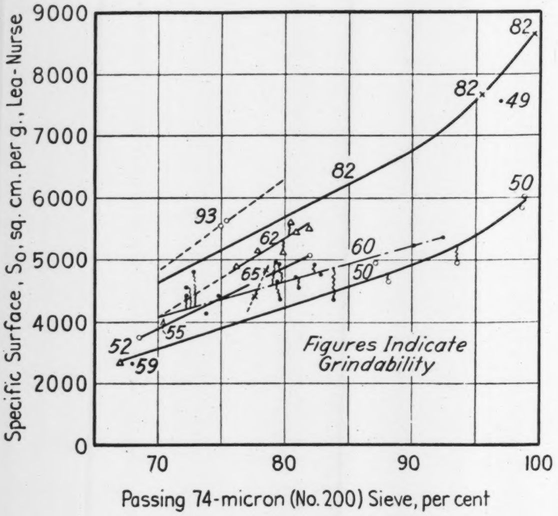

FIG. 12.—Relation Between Specific Surface and Percentage Passing 74-micron (No. 200) Sieve; "E" Series Pulverizer.

method of measuring surface area, are not the same as those obtained when following the principles of Method D 409.

While it is considered that the indices based on specific surface measurements are more accurate, this does not mean that such methods will have to be adopted for measurement of grindability. The curve of Fig. 10 indicates that there is a definite relation between the present indices and those based on specific surface. It may be that this curve will not hold for every individual sample but unquestionably it is as accurate as the sampling and other features that may enter into the determination of the grindability of coal, and it would seem as if this curve could be used as a conversion ratio.

Pulverized Coal:

In batch pulverizers, such as the grindability testing machine, where the minus 44-micron material may contain particles as small as 1 micron in diameter, specific surface data may be needed to amplify the sieve analysis. This condition is particularly true in the case of coals of high grindability.

In the case of continuous pulverizers, particularly air-swept pulverizers with classifiers, we have a condition where there may be a greater uniformity in particle size and under these conditions the relation between percentage passing a given sieve and specific surface may be quite uniform up to some limiting value.

The variation in the specific surface of the minus 44-micron material produced in the grindability testing machine led to the testing of a number of samples of pulverized coal in order to establish the relation between percentage passing a 74-micron (No. 200) sieve, specific surface, and grindability.

The data thus obtained were plotted and have been reproduced as Figs. 11 and 12.

To date sufficient data have not been accumulated to warrant the drawing of final conclusions but certain definite trends have been observed.

For any one type of pulverizer, the specific surface for any given percentage passing a 74-micron (No. 200) sieve increases with grindability.

The surface produced for a given percentage passing varies with the type of pulverizer and classifier.

The curves of Figs. 11 and 12 suggest that as the percentage passing decreases, the specific surface decreases, proportionately. On the other end of the scale there is a marked rise in surface as the percentage passing a 74-micron sieve approaches 100.

The point where the curve changes shape appears to be a function of grindability, occurring at lower percentages passing for high grindability coals than for low grindability coals.

There is little question but that a great deal of work remains to be done before final conclusions as to the effect of specific surface on grindability indices and the measurement of fineness of pulverized coal can be drawn.

The use of sieve analysis, particularly a 74-micron (No. 200) sieve, for measuring the fineness of pulverized coal is well established and in many instances it furnishes all the data required. The Lea-Nurse method of determining specific surface may not lend itself readily to plant control, and where extreme fineness is not required, its use may not be justified. For those operators, who, for one reason or another, require very fine grinding, this method of evaluating their finished product may find widespread use.

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(5) H. Heywood, "Measurement of the Fineness of Powdered Materials," *Proceedings*, Inst. Mechanical Engrs. (British), Vol. 140, p. 257 (1938).

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(6) J. Kozeny, "Über die Kapillare Leitung des Wassers Im Boden," *Berichte* der Wiener Akademie, Vol. 136a, p. 271 (1927).

(7) F. M. Lea and R. W. Nurse, "The Specific Surface of Fine Powders," *Journal*, Soc. Chemical Industry, Vol. 58, p. 277–283 (1939).

(8) Symposium on New Methods for Particle Size Determinations in the Subsieve Range, Washington Spring Meeting, Am. Soc. Testing Mats. (1941). (Issued as separate publication.)

(9) P. C. Carman, "Fluid Flow Through Granular Beds," *Transactions*, Inst. Chemical Engrs., Vol. 15, p. 150 (1937); "The Determination of the Specific Surface of Powders, Part I," *Journal*, Soc. Chemical Industry, Vol. 57, p. 225 (1938); "The Determination of the Specific Surface of Powders, Part II, *Journal*, Soc. Chemical Industry, Vol. 58 p. 1 (1939).

DISCUSSION

Mr. P. S. Roller[1] (*presented in written form*).[2]—A laboratory crushing device analogous to the impact tester employed by Romer was described in 1929 by Gross and Zimmerley[3] of the U. S. Bureau of Mines, who used it in careful measurements of the relationship between work input and surface produced in crushing quartz. They concluded that the relationship between work and surface is linear and that Rittinger's law is accurately obeyed. Their conclusion supports Romer's assumption regarding this relationship.

An impact crusher such as that described in Romer's paper seems very attractive as a means of estimating grindability in view of its simplicity and the fact that the work input is definitely known in foot-pounds or ergs. An obstacle that might remove the method from consideration would be nonconformance of the values it gives with the standard test, but the author has shown that it arranges the coals in the same order of grindability as the standard test and gives numerical values that are comparable. Grindability is a complicated phenomenon that depends on many conditions, as Romer's paper partly shows, and there is no *a priori* reason for favoring one set of results over any other set that differs from it only in moderate degree.

My own investigations in this field lead me to affirm that the Hardgrove grindability index expressed in terms of the percentage passing the No. 200 sieve is fundamentally sound, since it reflects fairly accurately the surface area of the material whenever, as in Romer's experiments, the percentage passing No. 200 sieve is not too great.

The air-permeability method of determining surface area (called in Romer's paper the Lea-Nurse method) without question is accurate within its field. I do not believe its field includes the ascertainment of submicroscopic irregularities, but for ascertaining geometrical irregularities the method apparently serves well. There is only one question and that is whether there is a limitation to the fineness of the sample. In the liquid-permeability method such a limitation of fineness was found, as stated on page 1154 of Romer's paper. In a comparison of results using other methods with those of air permeability,[4] there was evidence that when the heterodisperse powder was very fine the values obtained by the air-permeability method were low. As an explanation, it was suggested that channeling of the air might occur in a fine heterodisperse material. Further studies would be desirable on this point, as it may have some bearing on the lack of regularity in the ratios (surface area by air permeability to Wagner turbidimeter values) given in Table I of Romer's paper.

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[1] Physical Chemist, Eastern Experiment Station, U. S. Bureau of Mines, College Park, Md.

[2] Published by Permission of the Director, U. S. Bureau of Mines, Department of the Interior.

[3] J. Gross and S. R. Zimmerley, "Milling and Concentration," *Technical Publication 127*, Am. Inst. Mining and Metallurgical Engrs. (1929).

[4] P. S. Roller and P. V. Roundy, Jr., "Surface Area of Portland Cement," Symposium on New Methods for Particle Size Determination in the Subsieve Range, p. 36, Washington Spring Meeting, Am. Soc. Testing Mats. (1941). (Symposium issued as separate publication.)

MR. RALPH A. SHERMAN[5] *(presented in written form)*.—The many data that have been accumulated by the author on the relation of the work done on a sample of coal to the new surface produced prove quite conclusively that Rittinger's law is valid. Previous apparent inconsistencies in this relation when the number of revolutions or the load was increased were due merely to the lack of a method for the determination of the specific surface of the coal before and after grinding. The air permeability method fills this need; its application to the determination of the specific surface of pulverized coal would follow precedents established rapidly one after the other in the past two years in the cement, textile, and paint industries.

The paper furnished new data for use of Subcommittee VII on the Pulverizing Characteristics of Coal of the Society's Committee D-5 on Coal and Coke that may enable it to revise the present two tentative methods for determination of the grindability of coal. Inasmuch as the author has shown that the impact machine and the Hardgrove machine give comparable results when the air-permeability method is used for the determination of the new surface, it seems likely that the ball mill could also be used in a simpler and less tedious modification than the present method for the application of work to the sample of coal. Perhaps it may be possible to eliminate the specification of the type of machine from the standard and to specify only the method of measurement of the new surface produced and the method of expression of the relation to some standard coal.

If it were readily possible to define accurately the work done on the sample of coal, it should be possible to express the results merely in terms of new surface produced per unit of work and thus eliminate the reference to a standard of grindability such as the Emore coal. If no simple means are found to do this, the retention of the standard coal seems necessary and the author's method of calculation in which he makes the new index agree with the old in the range of 50 to 60 grindability, in which the majority of high-volatile coals fall, seems wise.

Because the apparatus is relatively simple and the time required for the determination of the specific surface, if the specific gravity is known, is so short, it would seem desirable to include the determination in a standard method rather than to refer to a conversion curve such as the author's Fig. 10. The apparatus and method should be of great value to any plant burning pulverized coal as a control of the product of their mills, for interpretation of the results with different coals, and as a guide to the adjustment of the air classifier on the mill.

MR. R. M. HARDGROVE[6] *(presented in written form)*.—The Lea-Nurse method of determining specific surface has provided a simple, rapid method of determining the specific surface of a pulverized product.

The determination of new surface produced in the grindability test by the Lea-Nurse method will increase the accuracy of the determination and should receive consideration by Subcommittee VII of Committee D-5 for revision of the Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 37 T)[7].

Figure 10 of the paper shows that the new surface produced as measured by the Lea-Nurse method is practically proportional to Method D 409 grindability throughout most of the range except for the higher grindabilities where it shows higher values. Figures 11 and 12 show the specific surface of pulverized coal to also increase rapidly at higher grindabilities for the same percentage passing a No. 200 sieve, which is consistent with the higher grindability values. This means that the relation between grindability and pulverizer capacity would not be altered appreciably if the grindability was determined by using the Lea-Nurse method to obtain the new surface, and the pulverizer output was based on the pulverized coal having a constant specific surface as determined by the Lea-Nurse method, that is, the same method of measurement used for the grindability test and for the testing of pulverizers. However, the adoption of specific surface as the unit of fineness in the testing of pulverizers would require considerable education, as the No. 200 sieve test is universally used at present.

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[5] Supervisor, Fuels Division, Battelle Memorial Institute, Columbus, Ohio.

[6] The Babcock & Wilcox Co., New York, N. Y.

[7] 1939 Book of A.S.T.M. Standards, Part III, p. 566.

There is also some question as to whether high specific surface in pulverized coal above certain values is justified, and perhaps the customary percentages passing a No. 200 sieve are really a better standard. This has been true in pulverizing cement rock where high surface does no good and the No. 200 sieve is still used as the best indication of satisfactory pulverization, although in the same plants specific surface is used as the standard for pulverizing cement clinker.

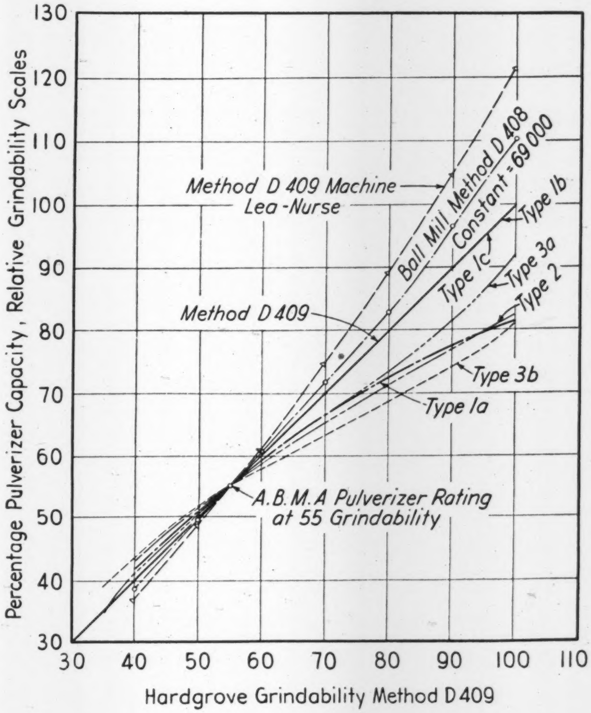

FIG. 1.—Relation Between Capacity of Various Types of Pulverizers and Grindability by Various Methods.

The accompanying Fig. 1 shows the relation between pulverizer capacity at a standard No. 200 sieve fineness and the Method D 409 grindability. This shows six types of pulverizers, two of which match the Method D 409 proportional line very closely, while the capacity of the other four falls below at high grindabilities. The two types matching the Method D 409 line have a very active circulation and prompt removal of fines and the finished product cushions the grinding process the least. The Method D 409 grindability scale represents the best pulverizer circulation developed so far.

The ball-mill grindability scale[8], when plotted against Method D 409, shows higher values at high grindabilities and the agreement with actual pulverizer capacities is not so good. Apparently Method D 408 is neither a true measure of the new surface or of relative pulverizer capacity at a standard No. 200 sieve fineness. The new scale obtained by using the Method D 409 machine, but determining the new surface by the Lea-Nurse method, is still higher and, therefore, more out of line with pulverizer capacity. If the Lea-Nurse method were used instead of the No. 200 sieve on the ball-mill test, the high grindability values would be higher than they now are.

As long as pulverizer performance is based on a standard No. 200 sieve fineness, it would seem very desirable to retain the Method D 409 scale of values. This is particularly desirable in view of the large amount of data available on the coals of the United States and the broad use by engineers of the Method D 409 scale in the purchase and testing of pulverizers.

It is our belief that the procedure that would result in the least confusion would be to use the Lea-Nurse method of determining the new surface in the Method D 409 machine and translating it to equivalent Method D 409 scale by a curve such as Fig. 10 of the paper.

MR. R. B. TEXTOR.[9]—In spite of all the work on this important determination of the grindability of coal, there still remains a question that to me seems to be bound up with the apparatus, and that is the fundamental definition. I am wondering whether Mr. Romer would say, in the light of his researches on the specific surface, whether he has anything to contribute in the way of a more fundamental definition of the grindability of a coal and of the index of grindability. There seems to be considerable confusion at present on this point in the minds of many people.

MR. J. B. ROMER[10] (*author's closure*).—Mr. Roller's very interesting comments on the relationship between work input and surface produced further substantiates the validity of the grindability testing machine as a device that obeys Rittinger's law.

Although we found the impact tester to be of great value in this particular research, we do not consider that such a machine would be desirable for the general determination of grindability.

Roller raises the question of whether there is a limitation to the fineness of the sample. Our data indicated that the results were reliable for materials that passed a 44-micron (No. 325) sieve. We did not endeavor to obtain data regarding the accuracy when the entire sample was smaller than 2 microns, which is the apparent limit for liquid permeability apparatus. We have measured the surface area of sublimed zinc oxide and obtained a value of the order of 18,000 to 19,000 sq. cm. per g. This represents an average particle diameter of less than 1 micron. No trouble was experienced in making these determinations but we do not have any data to substantiate the accuracy of the value obtained.

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[8] Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 – 37 T), 1939 Book of A.S.T.M. Standards, Part III, p. 562.

[9] President, The Textor Laboratories, Cleveland, Ohio.

[10] Chief Chemist, The Babcock & Wilcox Co., Barberton, Ohio.

I am sorry that I cannot furnish Mr. Textor with a new or better definition of the grindability of a coal and of the index of grindability.

I agree with Mr. Sherman as to the need for apparatus to measure the subsieve fractions and the desirability of applying such methods to many existing standards. In this connection, an article by Sherman[11] contains some interesting data on surface area which was determined by the tedious microscopic count method. Sherman's samples, which were prepared by grinding in a pilot plant mill, showed an entirely different surface area for a given sieve analysis than we obtained when measuring samples from production mills and which data were presented in Figs. 11 and 12. Sherman's figures do, however, show the same general relationship between grindability, the percentage passing a 74-micron (No. 200) sieve, and surface area.

Hardgrove brings up a very interesting point as to whether high specific surface in pulverized coal above certain values is justified. This is extremely interesting, since a study of Figs. 11 and 12 shows that for any given percentage passing a 74-micron (No. 200) sieve, the specific surface depends, among other things, upon the grindability of the material or, in other words, there is no fixed relationship between specific surface and percentage passing any given sieve.

The relation between the work input and the new surface produced is of interest to several of the discussors. Bond[12] makes the following statement:

> "It is interesting to note that about as much net energy is required to reduce a ton of quartz to particles of the grind-limit diameter by grinding as is required to melt the quartz. The two types of disassociation may be comparable in some respects."

Bond was dealing with three well-known western copper ores and for such material this relation may be more apparent than in the case of coal.

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[11] R. A. Sherman, "Burning Characteristics of Pulverized Coals and the Radiation from Their Flames," *Combustion*, December, 1933.

[12] F. C. Bond, "Measuring Surface Area in Grinding," *Technical Publication 1296*, Am. Inst. Mining and Metallurgical Engrs. (1941).

MEASUREMENTS OF POWER FACTOR AND DIELECTRIC CONSTANT AT ULTRA HIGH FREQUENCIES*

By K. G. Coutlee,[1] R. F. Field,[2] E. O. Hausmann,[3] Thomas Hazen,[4] and Harry R. Meahl[5]

Synopsis

The theory of the susceptance-variation method for measuring power factor at ultra high frequencies is discussed and four forms of apparatus are shown. The results of round-robin tests on solid dielectrics using these equipments are summarized. In addition, investigations of modifications of the resonant-circuit substitution method of the A.S.T.M. Tentative Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 150 – 39 T)[6] to adapt it to ultra high frequencies are reviewed, and both the negative results and valuable by-products of these investigations are given to aid anyone starting to make power-factor measurements at ultra high frequencies.

There have been many attempts made to extend to the ultra high frequencies the use of the well-known resonant-circuit resistance variation method for measuring power factor. However, lead inductance and series resistance effects became increasingly troublesome at the higher frequencies, tending to limit the range of this method and no apparatus or technique suitable for inclusion in the A.S.T.M. Tentative Methods D 150 was found. In 1936, a form of the resonant circuit susceptance-variation in which inductance and series resistance effects were reduced by having a parallel-plate variable air capacitor serve both as standard capacitor and sample-holder, was described (**1**).[7] Since then, four different forms of this have been built and used with good results. These equipments are shown in Figs. 1, 2, 3, and 4. Fundamentally, they all work on the same principle.

This paper presents the data taken in a series of round-robin tests by the authors using this circuit and forming the basis of the current proposed revision of D 150.

Theory

In the susceptance-variation method of measuring power factor, the change in conductance of a parallel tuned circuit, Fig. 5, caused by the insertion of a sample of dielectric material in the capacitive branch is measured in terms of capacitance change, capacitance, and voltage ratio. A mathematical proof of this is readily obtained by calculating the total conductance of the measuring circuit, first for the condition of the sample in place and then for that of the sample removed. The difference of these two conductances is the conductance of the sample, and the power factor is the ratio of this to the susceptance of the sample. It has been shown that:

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* The presentation of this paper at the annual meeting was sponsored by Committee D-9 on Electrical Insulating Materials.

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[4] Member of Bloomfield Laboratories, Bakelite Corp., Bloomfield, N. Y.

[5] General Engineering Laboratory, General Electric Co., Schenectady, N. Y.

[6] 1939 Book of A.S.T.M. Standards, Part III, p. 745.

[7] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 1182.

$$G_t = \frac{\omega \Delta C}{2\sqrt{(V_r/V)^2 - 1}} \text{ total conductance} \ldots (1)$$

where $\omega = 2\pi f$ and f = frequency, cycles per second,

ΔC = change in capacitance necessary to tune the cir-

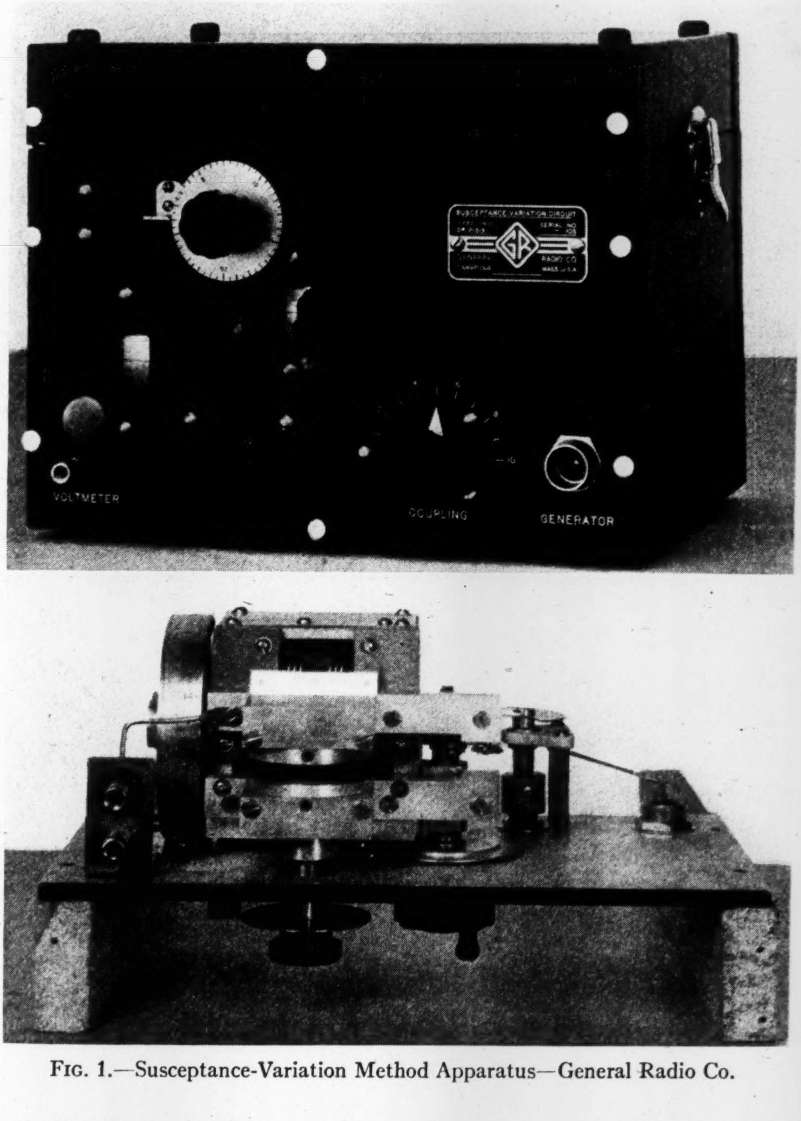

FIG. 1.—Susceptance-Variation Method Apparatus—General Radio Co.

cuit from V through V_r to V again.

V_r = voltage at resonance, and

V = chosen value of voltage off resonance (ordinarily 0.707 V_r to simplify calculations).

Therefore

$$G_x = \frac{\omega(\Delta C_1 - \Delta C_0)}{2\sqrt{(V_r/V)^2 - 1}} \text{ sample conductance} \quad (2)$$

where $\Delta C_1 = \Delta C$ sample in place, and

$\Delta C_0 = \Delta C$ sample removed

then

$$\tan \delta_x = \frac{G_x}{\omega C_x} = \frac{\Delta C_1 - \Delta C_0}{2C_x\sqrt{(V_r/V)^2 - 1}} \quad (3)$$

where C_x = capacitance of sample.

When $V = 0.707\ V_r$

$$\tan \delta_x = \frac{\Delta C_1 - \Delta C_0}{2C_x} \quad (4)$$

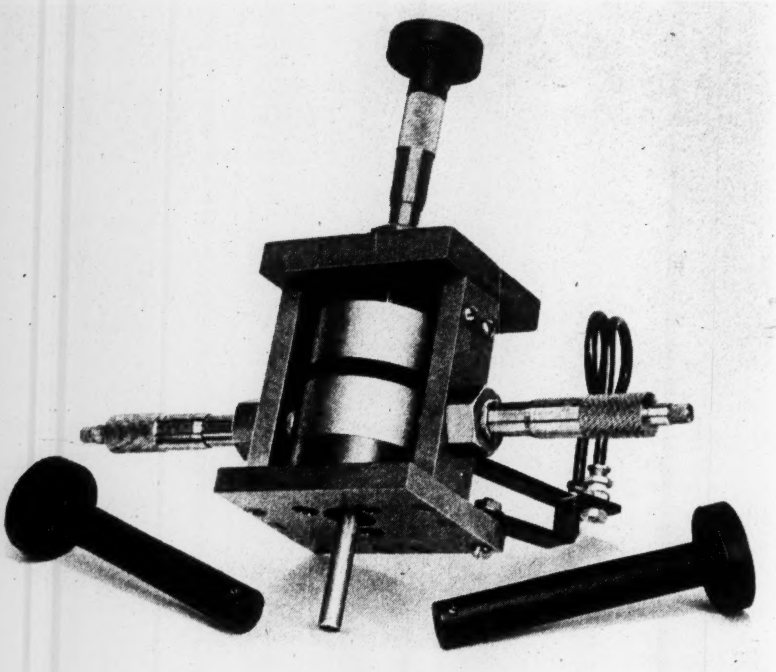

FIG. 2.—Susceptance-Variation Method Apparatus—Bell Telephone Laboratories, Inc.

When a parallel-plate capacitor is used as a combination sample holder and standard capacitor and electrodes completely cover the sample:

$$C_x(\mu\mu\text{f.}) = C_r(\mu\mu\text{f.}) \quad (5)$$

where C_r = geometric capacitance of parallel-plate capacitor, C_2, at the second resonance adjustment.

Taking the capacitance C_x of the test specimen equal to the geometric capacitance C_r of the parallel-plate capacitor automatically introduces a correction for fringing in the apparatus of Fig. 2 and gave a value within the accuracy requirements of D 150.[6] The following equation:

$$C_x = C_r - C_2' + C_a' \quad (6)$$

where C_r = calibration capacitance of parallel-plate capacitor C_2 at the second resonance adjustment,

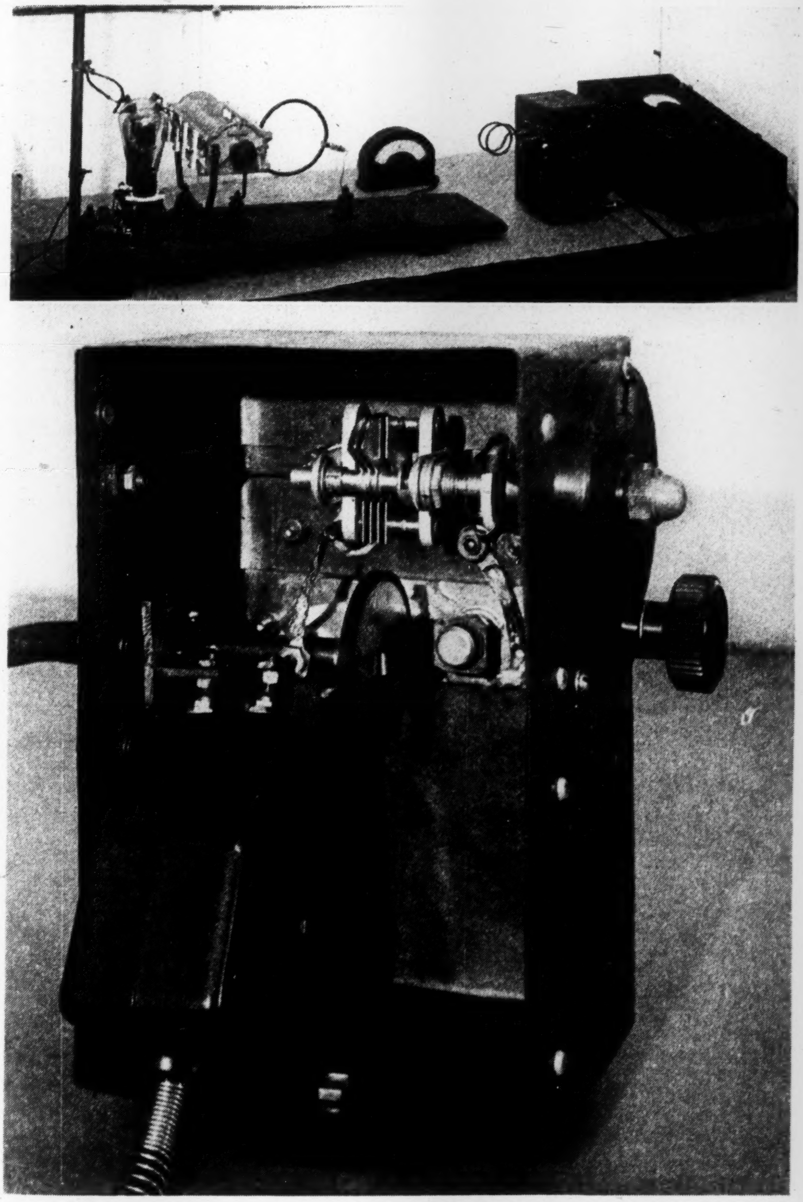

FIG. 3.—Susceptance-Variation Method Apparatus—Bakelite Corp.

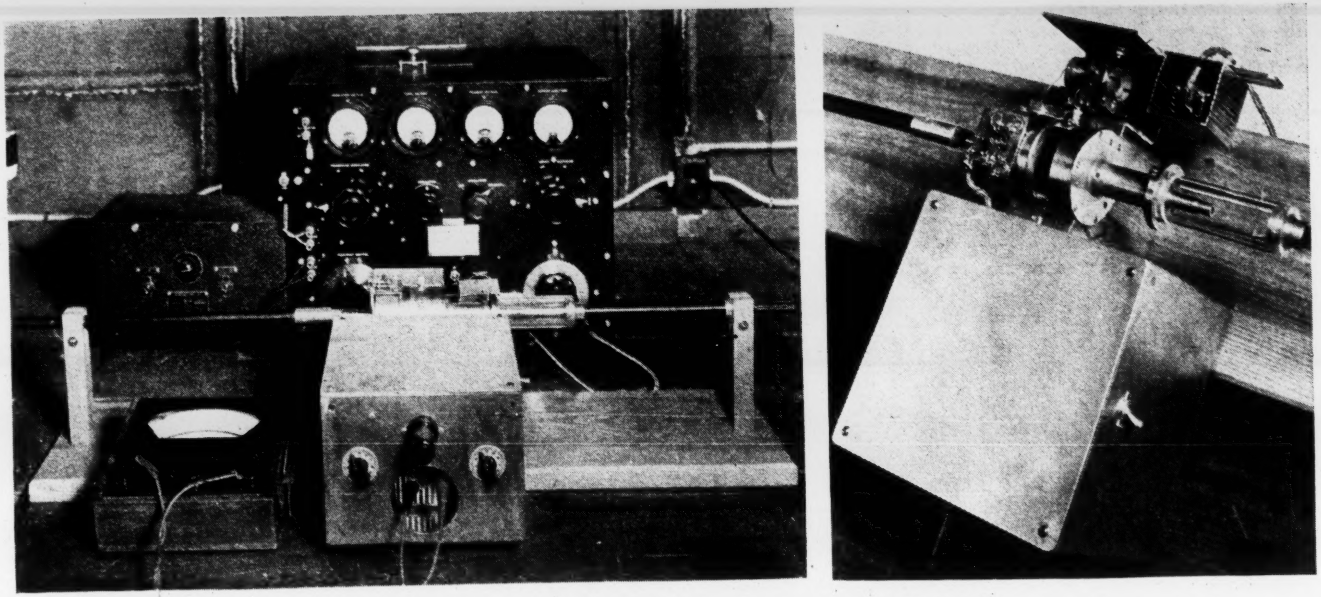

FIG. 4.—Susceptance-Variation Method Apparatus—General Electric Co.

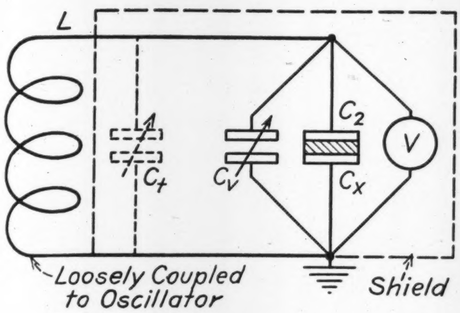

FIG. 5.—General Schematic Diagram of Susceptance-Variation Method.

C'_2 = calibration capacitance of C_2 at the setting with the sample in place, and

C'_a = computed capacitance of C_2 at the setting with the the sample in place,

was used with the apparatus of Fig. 1.

Since the resonance voltage when a sample is in the measuring circuit, V_1, is different from that when the sample is removed, V_0, and is inversely proportional to the conductance of the circuit,

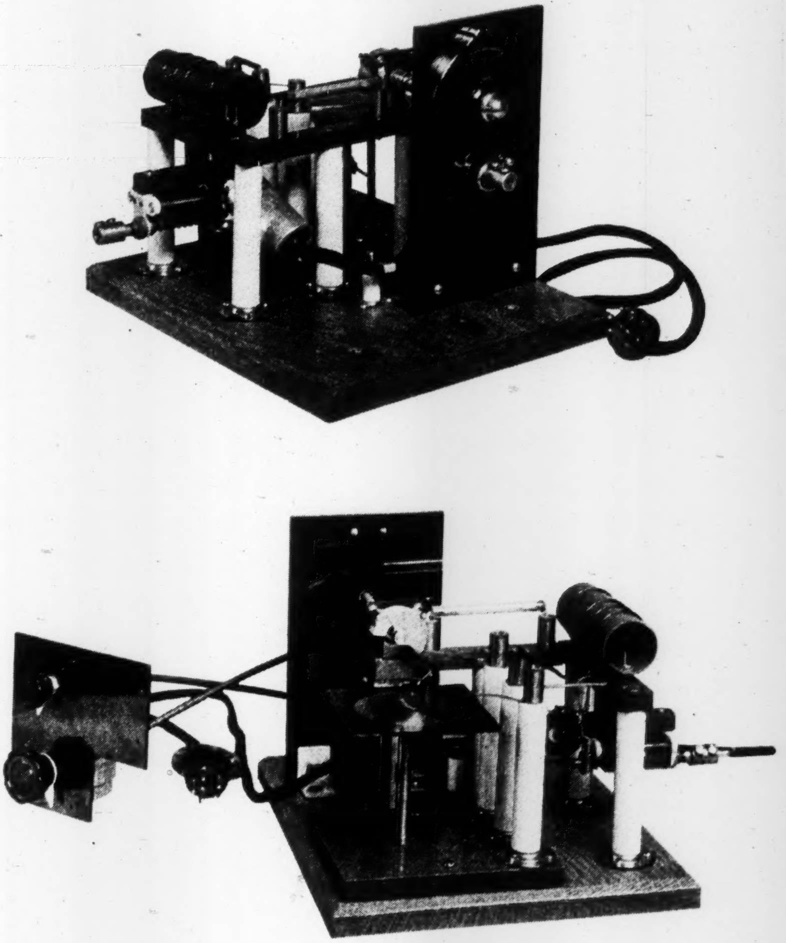

FIG. 6.—Resistance-Variation Method Apparatus—Continental Diamond Fibre Co.

the power factor may also be calculated from the following equation:

$$\tan \delta_x = \frac{\Delta C_x}{2C_x} \times \frac{V_0 - V_1}{V_1} \quad \ldots\ldots (7)$$

The dielectric constant may be calculated from the formula:

$$K = \frac{C_x}{C_a} \text{ where } C_a(\mu\mu\text{f.}) = 0.2249\, A/t \;.\; (8)$$

where t = average thickness in inches of test specimen excluding the thickness of specimen electrodes, and

A = area in square inches of specimen electrodes.

MODIFICATIONS OF D 150 – 39 T

Before the four different forms of the susceptance-variation method were built, several modifications of the resonant-circuit resistance variation method were attempted, a typical apparatus being shown in Fig. 6. The results were negative in so far as obtaining a method suitable for A.S.T.M. work was concerned. Valuable information, some of which became evident during this study and the preliminary work with the susceptance variation circuit is as follows:

1. The input to the measuring circuit must be stable with respect to both frequency and amplitude. Changes of 0.1 per cent are barely tolerable.

2. The measuring circuit must be completely shielded.

3. Ground connections must be short and massive.

4. The inductance used should have a Q (ratio of ωL to R) of at least 200. Increasing Q increases the sensitivity of the circuit.

5. The power factor or tan δ of the circuit should not exceed 0.01 and should be kept as small as possible.

6. The vacuum-tube voltmeter, or whatever indicating instrument is used, must have a negligible effect on the measuring circuit. In a vacuum-tube voltmeter, this means low input capacitance and conductance. The "acorn" diode vacuum tube is well suited for this purpose and other types have been found satisfactory.

7. Loose coupling between the source of radio frequency power and the measuring circuits is necessary, inductive, capacitive and resistive coupling being found equally satisfactory.

8. The geometry of the measuring circuit must remain practically constant with the sample "in" and "out" of the circuit.

9. The threads of the screw mounting and movable plate of the parallel-plate capacitor may require a constant impedance shunt consisting of a mercury well or flexible copper screen to promote stable electrical performance.

10. The fixed (high) plates of the parallel-plate capacitor and the vernier capacitor should be common if possible, while their movable plates should be effectively grounded.

11. The leads from the vacuum tube voltmeter to the fixed (high) plates of the parallel-plate and vernier capacitors should be short.

TEST METHODS

The same care that is exercised in preparing samples for power factor tests at low frequencies with regard to cleanliness, low resistance electrodes, drying, or other conditioning should be used when high-frequency measurements are made.

It is obvious from the nature of the test that considerable latitude is allowable in test methods. It was found that a sample of clear fused quartz may be used as a standard specimen to test any method. Due to its low power factor of 0.0002, which generally has been accepted as being constant over a wide frequency range, errors caused by any inductance residual in the equipment may readily be detected. Improvement particularly in the sensitivity of the equipment and technique possibly will lead to better agreement with the flat frequency characteristic of clear fused quartz than that shown in the curves.

One way of using the susceptance-variation method for measuring the power factor of solid dielectrics is to insert a specimen capacitor, with electrodes affixed, in the parallel-plate holder, C_2, and resonate the circuit at the desired frequency by means of C_t (alternatives are to vary L or the oscillator frequency). Note the maximum voltage, V_1, and then adjust the vernier, C_v, until the voltage is reduced to 0.707 V_1 first on one side, then on the other, of the resonance setting recording the capacitance values C_1 and C_1'. Return C_v to resonance setting. Remove the specimen and restore resonance by means of parallel-plate capacitor, C_2. Note the maximum voltage V_0 and record the capacitance value C_r of C_2. Again determine the change of capacitance to tune from 0.707 V_0 to 0.707 V_0 again by means of C_v and record C_0 and C_0'. Then $\Delta C_1 = C_1 - C_1'$ and $\Delta C_0 = C_0 - C_0'$ and tan δ may be calculated by using Eq. 4,

$$\tan \delta_x = \frac{\Delta C_1 - \Delta C_0}{2C_x}$$

Description of Equipment

When the parallel-plate capacitor, C_2, has 2-in. diameter electrodes, it has a capacitance range of from 7.07 $\mu\mu$f. at 0.100 in. separation to 70.7 $\mu\mu$f. at 0.010-in. separation. The movable plate is usually mounted on a fine thread, the conventional 1-in. machinists micrometer having 40 threads to the inch being found mechanically satisfactory.

Vernier capacitors on standard micrometer drives having capacitance changes per division of from 0.002 $\mu\mu$f. to 0.008 $\mu\mu$f. have been used.

In the form of the susceptance variation circuit shown in Fig. 1, shielding, terminals for a tuning coil, terminals for a vacuum tube voltmeter, terminals for a signal generator, a variable coupling capacitor, a variable calibrated tuning capacitor, a parallel-plate combination standard capacitor, and sample holder are built into a single unit. The vacuum tube voltmeter, tuning coil, and signal generator are not shown.

The data taken using this apparatus were analyzed by means of Eqs. 6 and 7.

Figure 2 shows a parallel-plate combination standard capacitor and sample holder with two micrometer vernier capacitors built in and a tuning coil attached. The signal generator, vacuum tube voltmeter, and coupling unit are not shown. The data taken using this apparatus were analyzed by means of Eqs. 4 and 5.

Figure 3 illustrates inexpensive construction and loose coupling. The data taken using this apparatus were analyzed by means of Eqs. 6 and 7.

Figure 4 shows a complete equipment consisting of a parallel-plate combination standard capacitor and sample holder, a micrometer vernier capacitor, a vacuum tube voltmeter, a tuning variometer, a signal generator, and an electrostatically shielded, resistance damped, inductive coupling unit. The eleven desirable features discovered while attempting modifications of D 150 were incorporated in this apparatus. The data taken using this apparatus were analyzed by means of Eqs. 4 and 5.

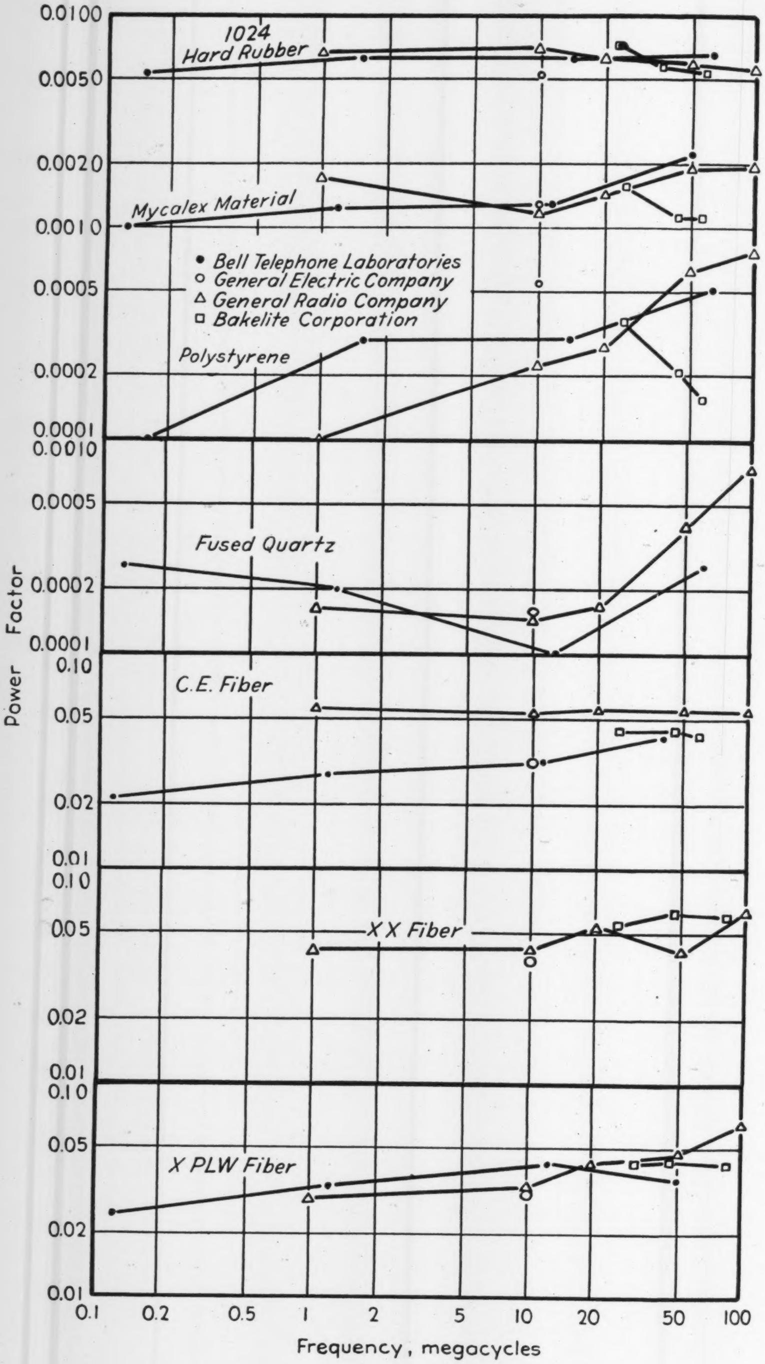

FIG. 7.—Power Factor *versus* Frequency Data.

TEST DATA

The test data obtained with samples of fused quartz, polystyrene, mycalex insulation, No. 1024 hard rubber, Grade CE natural phenol fabric, grade XX natural phenol fiber, and grade XPLW natural phenol fiber in a round robin by Section G on Measurement of Power Factor at Ultra High Frequencies of Subcommittee VII on Electrical Tests of Committee D-9 on Electrical Insulating Materials are shown graphically in Figs. 7 and 8.

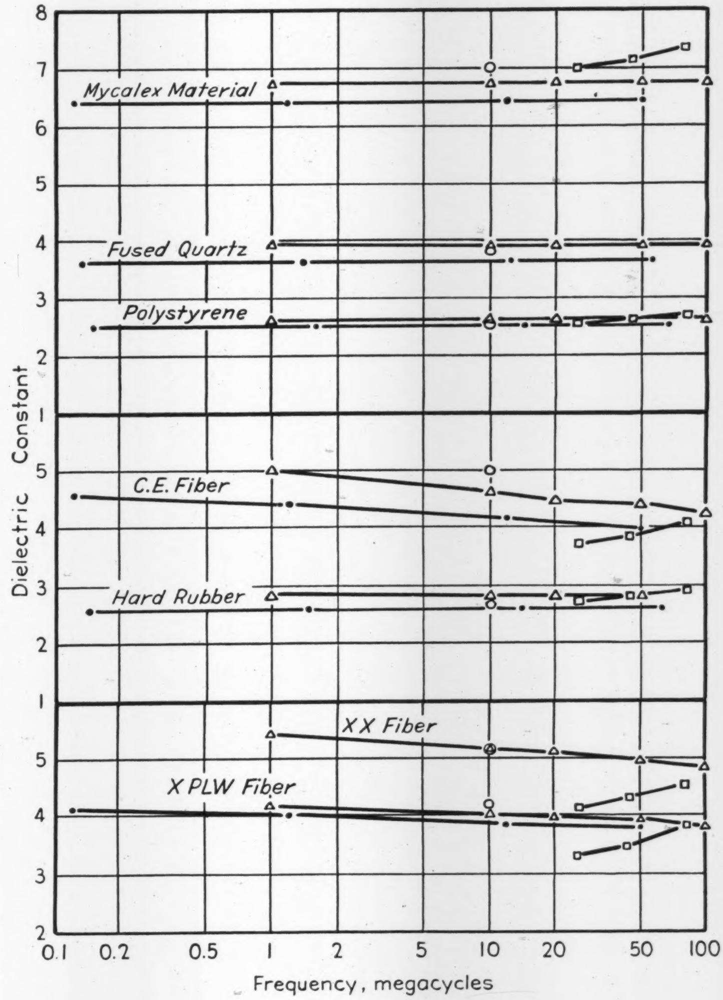

FIG. 8.—Dielectric Constant *versus* Frequency Data.

DISCUSSION OF DATA

The data were taken at room temperatures and humidities, on different sets of samples, over a period of about four years and using apparatus differing as shown in Figs. 1, 2, 3, and 4. The agreement obtained is considered satisfactory for A.S.T.M. purposes. The departure from a flat frequency characteristic for fused quartz indicated by the curves apparently is caused by a high experimental error, because the low power factor values of quartz approach the limitations of the method.

The data presented graphically show that the susceptance-variation method of measuring power factor is suitable for A.S.T.M. work over the power factor range of 0.0001 to 0.1 and over the frequency range of 0.1 to 100 megacycles per second.

REFERENCES

(1) L. Hartshorn and W. H. Ward, "The Measurement of the Permittivity and Power Factor of Dielectrics at Frequencies from 10^4 to 10^8 Cycles per Second," *Journal*, Inst. Electrical Engrs., Vol. 79, pp. 597-609 (1936).

(2) D. B. Sinclair, "Impedance Measurements at High Frequencies with Standard Parts," *General Radio Experimenter*, Vol. XIV, No. 4, September 1939, pp. 1-8.

(3) P. C. Michel, M. W. Scheldorf, and T. M. Dickinson, "Measurements at Radio Frequencies," Harry R. Meahl, *Electrical Engineering*, Vol. 59, No. 12, December, 1940, pp. 654-660.

(4) Von Lothar Rohde, "Measurements of Dielectric Loss at 2 to 5×10^8 Cycles Per Second," *Zeitschrift für Technische Physik*, Vol. 16, pp. 637-639 (1935).

REPORT ON ROUND-ROBIN TESTS OF POWER FACTOR AND DIELECTRIC CONSTANT FOR GLASS*

By Philip A. Richards[1]

Synopsis

Recognizing the need for better coordination of test methods, Section B of Subcommittee V of the Society's Committee D-9 met in Corning in 1935 to organize a study of this problem. It was decided that each member of the committee who was in a position to make measurements would undertake the problem independently and independent of any other set of standardized measurements.

From five commercial glasses representative of high and low-loss types 60 samples were prepared. The samples were circulated in a "round-robin" manner to six laboratories. The data from all of the laboratories were plotted on one curve for each type of glass.

Conclusions drawn from these data were that for accurate power-factor measurements it was necessary to eliminate stray circuit components so far as possible and to evaluate correctly those remaining. Further, the increase in power factor which has been noted when the substitution method is used at higher frequencies is almost certainly due in part to resistance in the leads to the test condenser.

The substitution method for measuring power factor described in A.S.T.M. Methods D 150 – 36 T is satisfactory for glass when minor modifications are made. Recommendations have been made to modify the standards in the light of the data presented.

Large discrepancies have been found in dielectric constant and power factor measurements made on glass by different observers in different laboratories.[2]

Recognizing the need for better coordination of test materials, Section B on Glass of Subcommittee V on Ceramic Products (Porcelain, Glass, Etc.) of Committee D-9 on Electrical Insulating Materials met in Corning, N. Y., in 1935 to organize a study of this problem. It was decided that each member of the committee who was in a position to make measurements would undertake the problem independently and independent of any other set of standardized measurements. After a satisfactory degree of progress had been made, the members were to reassemble, compare their results and agree upon the best technique to be followed. From this technique it was thought to be possible that specifications for making measurements on glass could be drawn up.

Comparisons of test methods indicated that each laboratory employed different technique in their measurements. The circuits used were different as were the treatments of the samples prior to test. To eliminate the possibility of variation of samples, it was decided that the same samples should be tested by all in a "round-robin" manner. Since each laboratory had experience in the use of its particular methods it was decided that each should use the ones ordinarily employed. This was to apply to test circuits, sample handling, electrode application, etc. Two exceptions to this were made: one consisted in the application of permanent electrodes to one group of the samples, and the other was the use of a sample for test circuit check to be circulated with the regular samples.

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* The presentation of this paper at the annual meeting was sponsored by Committee D-9 on Electrical Insulating Materials.

[1] Research and Engineering Department, RCA Manufacturing Co., Inc., Harrison, N. J.

[2] For a more detailed discussion see J. T. Littleton and G. W. Morey, "Electrical Properties of Glasses," p. 122, John Wiley and Sons, Inc., New York, N. Y.

The permanent electrodes were suggested because some tests had indicated that the method of applying the electrodes and particularly the nature of the adhesive used was the cause of variation in results. It was felt that the contact obtained with a metal film deposited at elevated temperatures provided more intimate contact and that this method might be preferable. After all of the data were gathered, we planned to choose the methods giving most reliable results consistent with ease of manipulation and simplicity of equipment. The results of the findings were to be presented to Committee D-9 and to the Society for inclusion in the Society's standards.

Standard disk-type samples of glass with known histories were, therefore, prepared at Corning. In addition a special "sandwich sample" was made with heavy platinum electrodes sealed into the glass under vacuum to exclude bubbles. The "sandwich" was completely embedded in a glass block. It was further improved at the Bell Laboratories by sealing glass tubes over the platinum lead-in wires to eliminate surface leakage.

The power factor *versus* frequency curves obtained by several observers were found to have a relatively flat portion at the lower frequencies and then to bend upwards at the high frequency end. This effect appeared at different frequencies in the several laboratories. Further there seemed to be no theoretical basis for explaining the effect. We planned a study of the results of the larger group cooperating in this test to see whether the discrepancies might be attributed to:

(*a*) True dielectric properties of the glass.

(*b*) Phenomena connected with the measuring apparatus.

(*c*) The nature of the contact between the electrode and dielectric.

PROCEDURE

For use in the round-robin measurements five commercial glasses were selected with electrical properties representative of the range of values likely to be encountered in practice including high-loss glasses not primarily intended for electrical purposes, as well as very low-loss glasses developed for electrical and radio service. Each type of glass was given ordinary production annealing in accordance with the Corning schedule appropriate to the glass type. Sixty samples were prepared from these five representative glasses in the form of approximately $\frac{1}{8}$ by 6-in. disks and $\frac{1}{8}$ by 2-in. disks.

Six 6-in. samples of each of the five different glasses were included of which three had fired-on silver electrodes; the rest were blank for whatever type of electrodes the different laboratories preferred to use. A set of six $\frac{1}{8}$ by 2-in. disks were included for such of the laboratories as were equipped to make measurements at the very high frequencies. Each sample was marked with a diamond for identification in accordance with the following code:

Glass Composition	6-in. Disks	2-in. Disks
Low-alkali borosilicate for high-frequency electrical service	A_1 to A_6	A_7 to A_{12}
Borosilicate with high chemical durability	B_1 to B_6	B_7 to B_{12}
High lead glass	C_1 to C_6	C_7 to C_{12}
Ordinary lead glass	D_1 to D_6	D_7 to D_{12}
Lime glass	E_1 to E_6	E_7 to E_{12}

SUMMARY OF TEST PROCEDURES

It is impractical to include all of the procedure details reported by the individual laboratories. Each employed its own variations of similar methods or entirely different ones. The pertinent features of each will be outlined, however, so that the conclusions in this report as to their relative merits may be weighed.

Corning Glass Works:

The measurements were made by a resonance substitution method using a sensitive vacuum tube voltmeter with a mirror galvanometer in the plate circuit as a resonance indicator. The frequency used was 760 kc. and the temperature 25 C. Readings were made without correcting for losses in the standard condenser, since their purpose was merely to check the range of power factor rather than secure absolute values. It is interesting to note that the readings fall within the spread of the composite data.

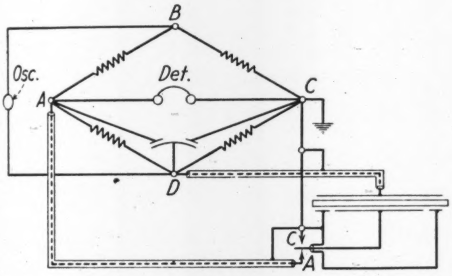

FIG. 1.—Triple Shielded Bridge. Shielding not shown.

Bell Telephone Laboratories, Inc.:

The frequency range of 70 cycles to 5 megacycles was covered in these measurements. Two separate completely shielded capacitance and conductance bridges (parallel resistance type) were required to cover this range: one from 70 cycles to 100 kc. and another of special high-frequency design from 100 kc. to 5 megacycles.

A schematic diagram of the low-frequency range bridge minus shielding is shown in Fig. 1. The high-frequency bridge was much the same except for a balanced lead switching arrangement using a miniature double-pole, double-throw mercury switch and an external shielded precision air capacitor from which the capacitance of the sample was obtained. Two bridge balances were required by both bridges for each measurement. The capacitance of the sample first was connected across the *CD* arm and then transferred by means of a switch to the *AD* arm of the bridge as shown in Fig. 1, direct capacitance[3] and conductance values thus being obtained.

The test frequencies were furnished by suitable vacuum tube oscillators. Vacuum tube detector amplifiers were used for the various frequency ranges. Bridge balances were indicated visually by means of a meter at the lowest and highest frequencies, and audibly by means of a telephone receiver through the audio frequency up to 100 kc., a heterodyne circuit, of course, being necessary above the audio range.

Guarded electrodes were used on both the regular and fired-on silver electrode samples. In the first case the electrodes and guard rings were tinfoil held in place by a small quantity of petrolatum. In the second case tinfoil guard rings were applied with petrolatum to the space outside the fired-on electrode. The use of guard rings eliminated fringing; therefore, corrections for edge effects in the calculation of dielectric constant were unnecessary.

RCA Manufacturing Co., Inc.:

The frequency range of from 0.3 to 17 megacycles was covered by the substitution method. The circuit recommended in the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 36 T)[4] was followed except that the known resistance was placed in series with the standard condenser and the switching was arranged to include either the sample or the standard condenser and resistor rather than the parallel combination. The vacuum tube voltmeter method of indication was employed. For frequencies above 1 megacycle the resistors consisted of short straight sections of resistance wire. For lower frequencies a good-grade decade resistance box was employed. All tests were made at room temperature.

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[3] G. A. Campbell, "Direct Capacity Measurements," *Bell System Technical Journal*, July, 1922, pp. 18–38.

General Electric Co.:

The frequency range from 1 to 10 kc. was covered on a series resistance and capacitance bridge. The bridge was first checked with a fused quartz sample of known power factor. Clear fused quartz has been found useful for checking any type of measuring circuit since the power factor is constant over the 1 to 3000 megacycle frequency range. On a sample having a known value of 0.00022 the accuracy obtainable is ±0.00005.

The bridge was balanced with the sample out. The sample was then connected across the standard and the bridge rebalanced. Power factor was calculated from the readings obtained. Edge corrections for dielectric constant were obtained from measurements made on the bridge with and without guard rings applied to the sample. Edge corrections obtained on the bridge in this manner were applied to calculations for the higher frequencies also.

The frequency range from 100 to 1000 kc. was covered in a substitution circuit similar to that of Methods D 150 - 36 T[4] except that the switch is arranged to connect either the sample or the standard across the circuit. A thermocouple and galvanometer were used for indicator.

Tests were made at about 25 C. and 20 per cent relative humidity.

Owens-Illinois Glass Co.—Purdue University:

The frequency range from 1 to 1000 kc. was covered on a bridge of the type shown in Fig. 2. Careful arrangement of parts and shielding were employed to maintain constant capacitance to ground. The guard ring was connected as shown so its capacitance to ground would not shunt the standard. The sample is connected across the standard so at balance the change in capacitance with and without the sample represents the sample capacitance. The power factor at the low frequencies was determined from the change in resistance in the "balance" arm of the bridge: necessary to balance the bridge when the sample is added. At 300 kc. this change became too small to be measured accurately so the condenser *PF* was used. It was calibrated directly in terms of the power factor of the standard arm.

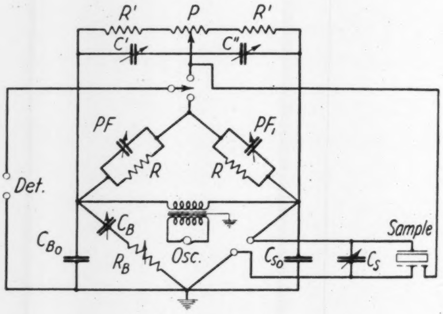

FIG. 2.—Bridge Circuit.

Edge corrections were taken from A.S.T.M. Methods D 150 - 36 T.[4]

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[4] Tentative Methods of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (D 150 - 36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1938 Book of A.S.T.M. Tentative Standards, p. 1071.

Measurements were made at 27.5 C. Humidity was lowered for the low-frequency tests only.

Westinghouse Electric and Manufacturing Co.:

The low-frequency measurements covering a range from 60 to 20,000 cycles were made on a capacitance bridge. Sufficient accuracy could not be obtained with direct-reading methods so a substitution method was applied to the bridge.

The measurements up to a million cycles were made in an ordinary resonant circuit, A.S.T.M. Methods D 150 – 36 T,[4] by the substitution method.

THE COMPOSITE CURVES

Graphical presentation of data was followed throughout the tests. As data were accumulated, meetings were held to discuss the progress and the plans for obtaining the best results from the tests still to be made.

Space is not available to show all of the curves. Some conclusions drawn from study of the curves are given and representative curves illustrating these points are presented (Figs. 3 to 11).

All values have been corrected for edge effect,[5] except where specifically noted and in the case of the "sealed-in" sample where no corrections are possible.

Data taken on the samples having silver electrodes were plotted separately from those taken with foil electrodes so that comparisons could be made.

Dielectric-Constant Curves:

On these curves the depressed-zero method of increasing spread is used. The glasses vary in dielectric constant from about 3.9 for type A to around 8.7 for type C. No general trend as denoted by the spread may be seen between glass types at low frequencies. It is noted, however, that the readings as taken on the bridges appear to agree better at low frequencies than at high frequencies. Furthermore, the readings as taken by the substitution method at higher frequencies have still greater spread.

Figure 3 illustrates the spread on the type A low-loss glass. Figure 4 shows the same glass when silver electrodes are used. The spread is seen to be nearly the same and the values also agree well. Therefore, we deduce that either silver or foil electrodes are equally suitable.

Figure 5 shows data on the high-loss type E glass. The fall from 8.2 to about 7.2 is interesting. These readings were taken on the same equipment as used for those of Figs. 3 and 4 so it is reasonable to attribute the difference to the material rather than to the methods of measurement.

Any increase of capacitance with frequency for any glass should be regarded as indicating too great inductance in the leads to the test condenser. When the substitution method is used in which the standard and test condensers are alternately switched into the circuit, the equality of inductance in the respective lead wires should be tested by measuring the capacitance of a sample known not to change with frequency.[6] For the substitution method in which the test condenser is added in parallel with the standard condenser, the inductance of the leads to the test condenser should be accurately determined by measuring a condenser which can be connected alternately at the ends of the leads and at the terminals of the standard condenser. This value of inductance is then used to correct the observed values of capacitance.

Power Factor Curves:

Figure 6 shows the composite curve for foil electrodes on type A glass. The

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[5] A. H. Scott and H. L. Curtis, "Edge Correction in the Determination of Dielectric Constant," *Journal of Research*, Nat. Bureau Standards, Vol. 22, June, 1939, p. 747.

[6] J. G. Miller, "A Method for Calculating the Effect of Residual Inductance in High Frequency Measurements," *Journal*, Am. Chemical Soc., Vol. 60, p. 42, January, 1938.

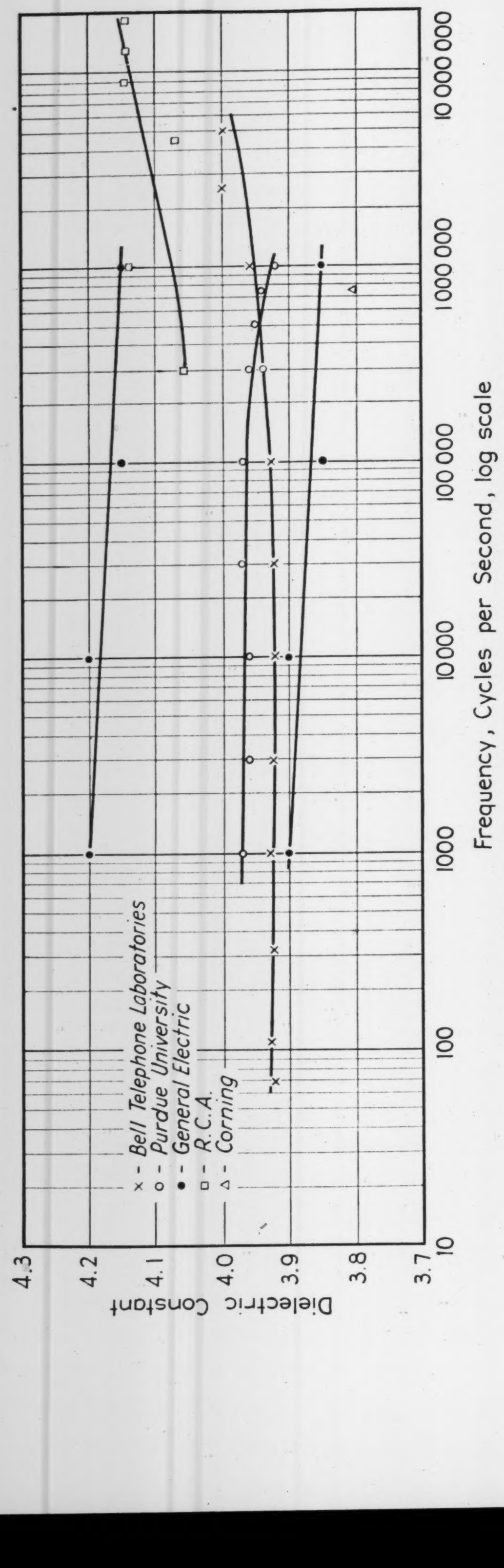

FIG. 3.—Dielectric Constant-Frequency Curves for Type A Glass—Foil Electrode.

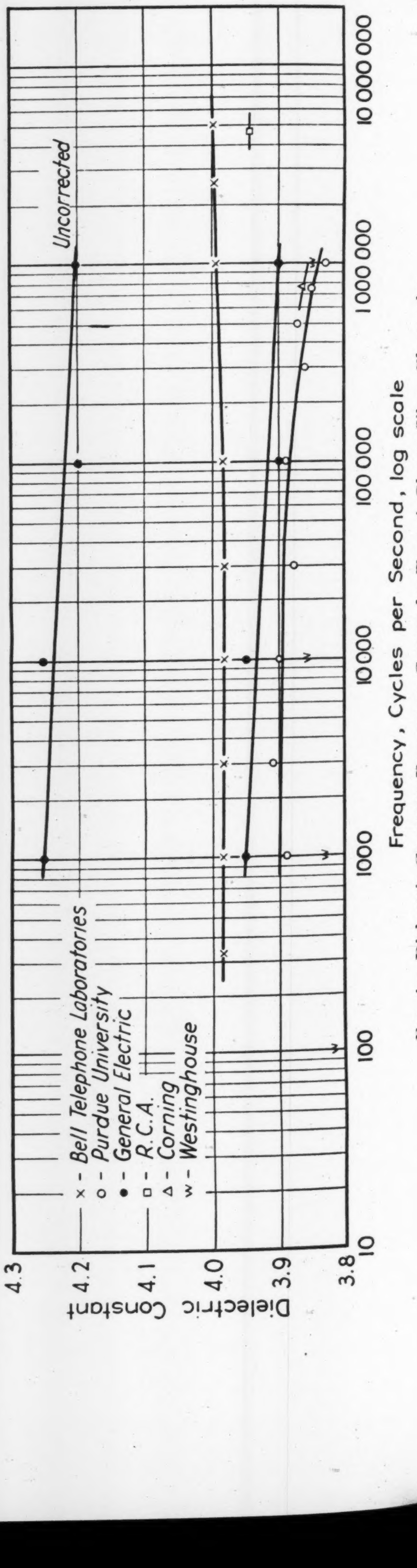

FIG. 4.—Dielectric Constant-Frequency Curves for Type A Glass—Silver Electrodes.

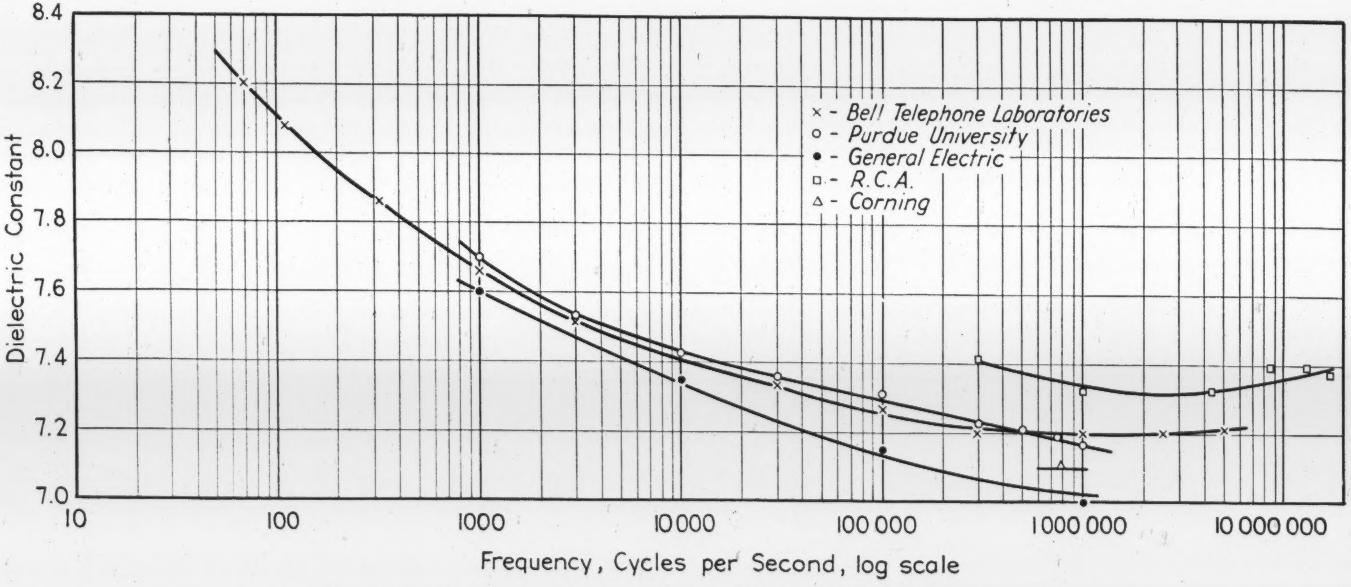

FIG. 5.—Dielectric Constant-Frequency Curves for Type E Glass—Foil Electrodes.

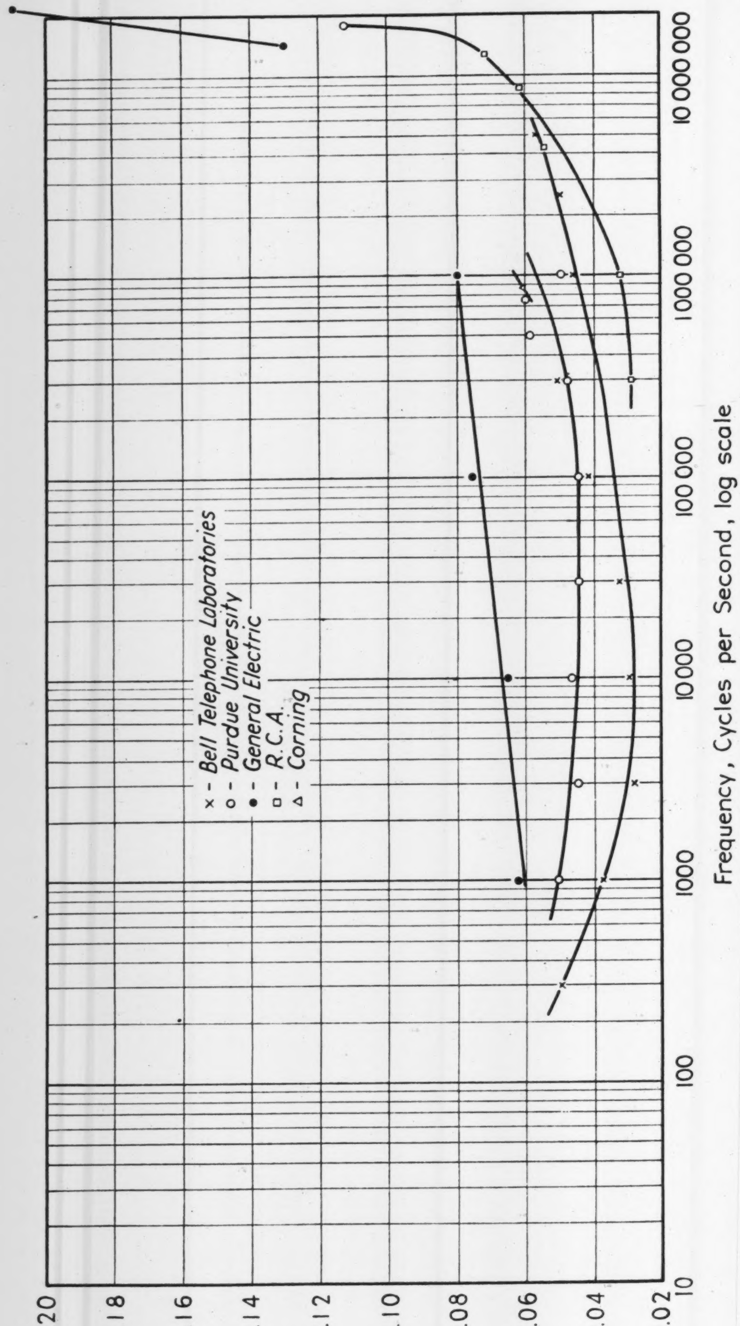

FIG. 6.—Power Factor-Frequency Curves for Type A Glass—Foil Electrodes.

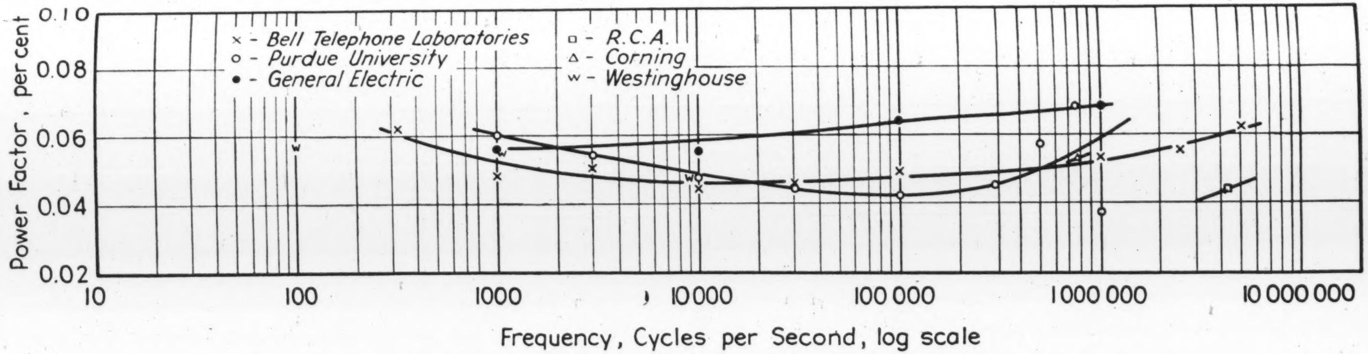

FIG. 7.—Power Factor-Frequency Curves for Type A Glass—Silver Electrodes.

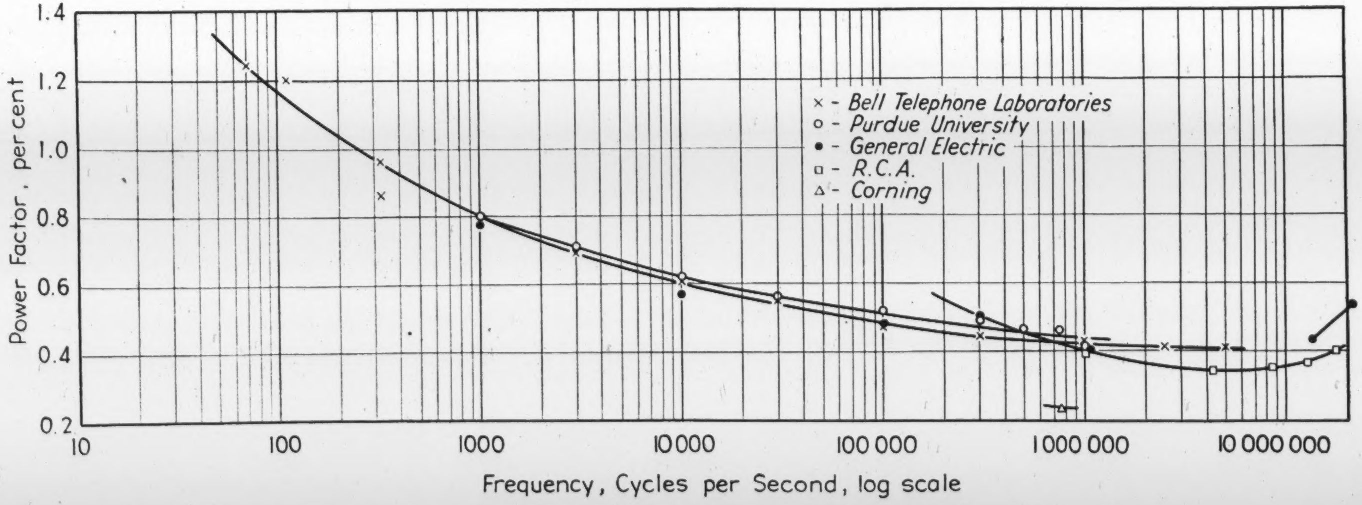

FIG. 8.—Power Factor-Frequency Curves for Type B Glass—Foil Electrodes.

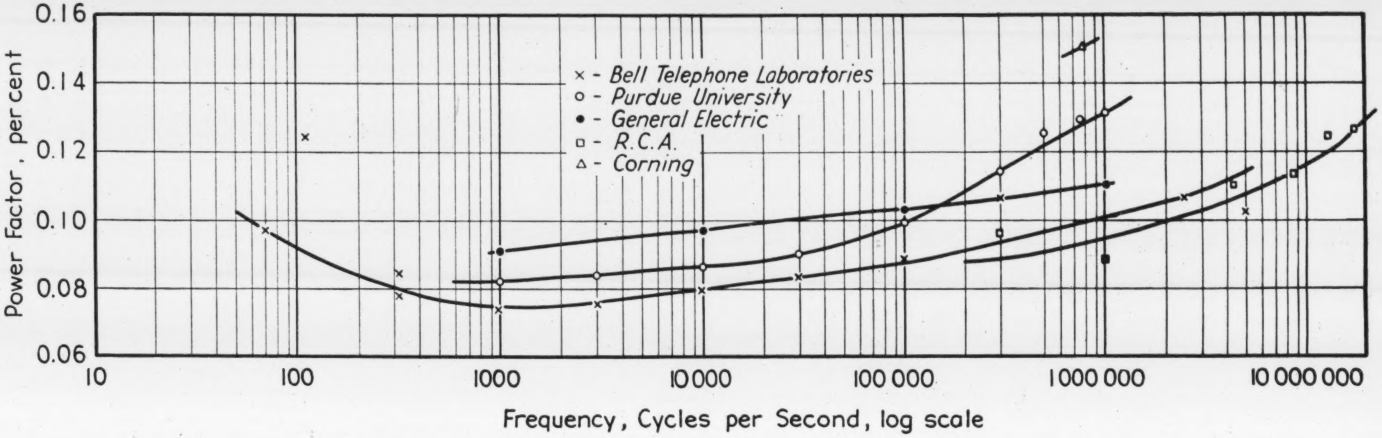

FIG. 9.—Power Factor-Frequency Curves for Type C Glass—Foil Electrodes.

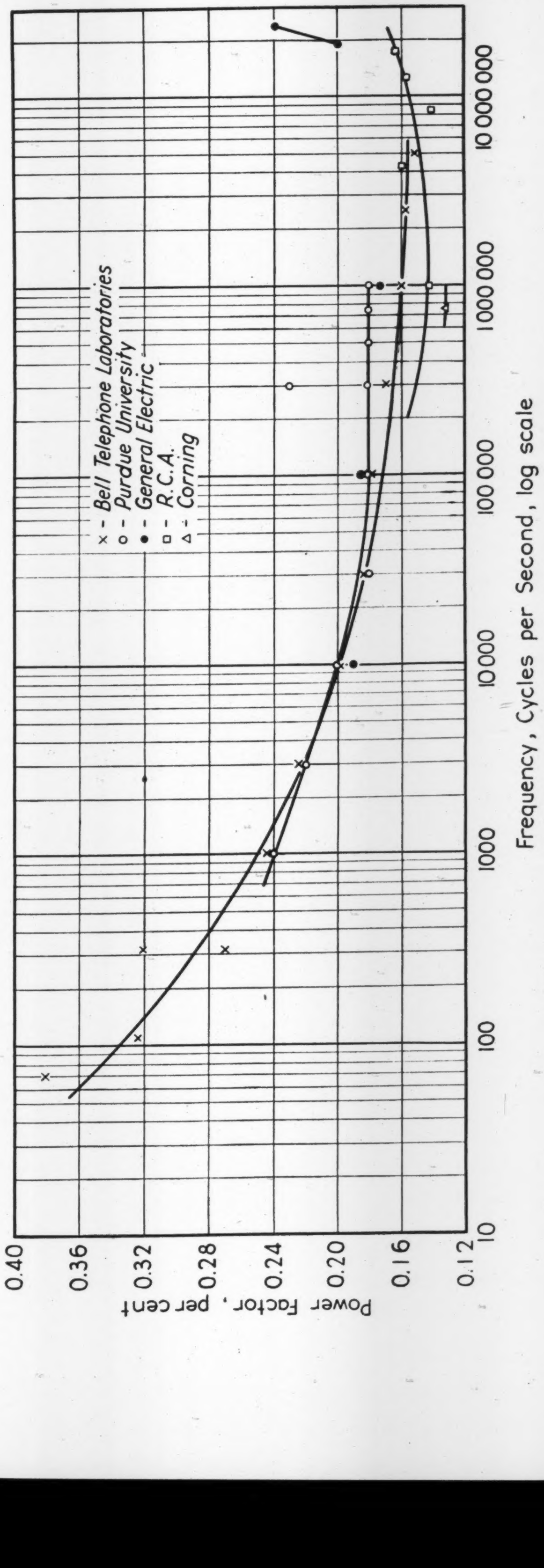

FIG. 10.—Power Factor-Frequency Curves for Type D Glass—Foil Electrodes.

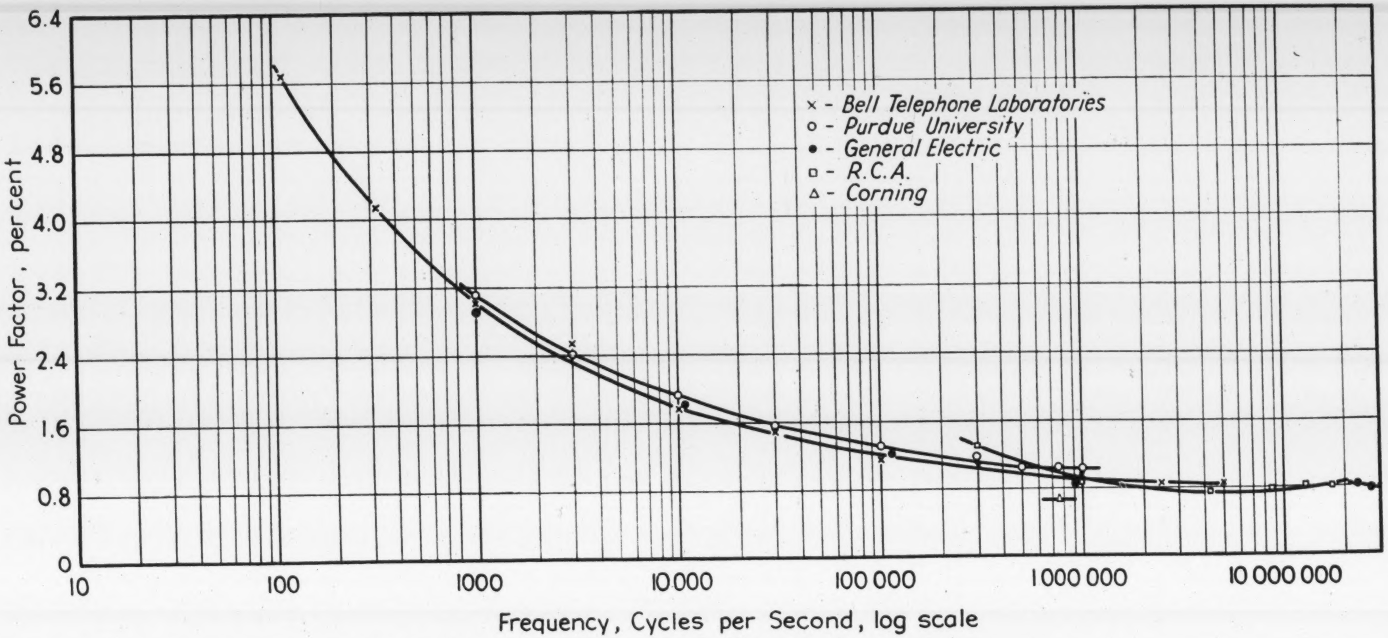

FIG. 11.—Power Factor-Frequency Curves for Type E Glass—Foil Electrodes.

spread of these curves is large. Comparing these data with those of Fig. 7 it is seen that while the spread is not as large when silver electrodes are used the curves appear to group near higher values than when foil is used. It had been noted that the coverage of silver was not entirely complete so the higher losses with silver may have been due to increased resistance of the electrode.

The increase in power factor at high frequencies is almost certainly due in part to resistance in the leads to the test condenser. When the standard and test condensers are alternately switched into the circuit, the test for equality of lead inductance is usually also adequate for equality of lead resistance, provided the pairs of leads are identical. When the test condenser is added in parallel with the standard condenser, the measurement of lead inductance also allows the determination of lead resistance. For both of these methods series resistance in the standard condenser will cause the value obtained for power factor to be low.[7]

Some laboratories in cooperation with Section G on Measurement of Power Factor at Ultra High Frequencies of Subcommittee VII on Electrical Tests of Committee D-9 are working on an ultra high frequency power factor method in which it is hoped the errors of lead inductance, etc., may be eliminated.[8] This work is progressing satisfactorily and it is expected that a new test method for frequencies up to 100 megacycles will be available shortly for inclusion in A.S.T.M. Method D 150.

The tendency of the power factor curves to rise at the high frequency end is less as the losses of the glass increase. This is shown in Fig. 11 which is the composite curve for the high-loss type E glass. Where the losses are as high as these the errors appear small.

THE "SEALED-IN" CELL

Power factor and dielectric constant data on the "sealed-in" cell were less consistent than data taken on glass in the usual manner. It was hoped that a suitable "sample" of this sort could be found for use in checking between laboratories. These data indicate that this particular construction is not reliable although some form of check sample would be a very useful tool.

DISCUSSION OF TESTS

Application of Electrodes:

Laboratories testing all of the samples of any one glass reported that the uniformity of the samples for each grade of glass was enough better than the errors of measurement so that lack of uniformity of the samples could be dismissed as a source of disagreement.

Foil electrodes were considered practical both for the ease of handling and the accuracy of the results. The foil electrodes are used with petrolatum as the adhesive. It is necessary to guard against contamination of the petrolatum. Moisture in the petrolatum has been found to influence low-frequency measurements. Of course, a minimum of the adhesive is essential and sufficient pressure and rubbing to force the foil into very intimate contact with the glass surface is found desirable. During the application of the electrodes care should be taken to minimize contamination of the surface not covered with foil so as not to nullify the results of the careful cleaning prior to this operation. The effects of leakage around the edge are most noticeable at low frequencies.

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[7] R. F. Field and D. B. Sinclair, "A Method for Determining the Residual Inductance and Resistance of a Variable Air Condenser at Radio Frequencies," *Proceedings*, Inst. Radio Engrs., Vol. 24, February, 1936, p. 255.

[8] L. Hartshorn and W. H. Ward, "The Measurement of the Permittivity and Power Factor of Dielectrics at Frequencies from 10^4 to 10^8 cycles per second," *Proceedings*, Wireless Section, Inst. Radio Engrs. (London), Vol. 12, March, 1937, p. 6.

The Test Circuit:

The most common source of error at radio frequencies is usually traceable to inductance or capacitance inherent in a system where several components are connected to form the test circuit. Accurate results can hardly be expected unless these sources of error are made negligible or their presence accounted for in the final calculations. For example, the method of switching either the sample or the standard into the substitution circuit has been recommended purely because it is often easier to make the leads symmetrical, and hence, have the errors cancel when this method is used. The errors due to losses in the standard are present when this method is used however. When the standard is left in the circuit, its dielectric losses do not enter into the calculations and only the losses caused by series resistance need be allowed for. In the latter case, however, it is physically impossible to connect the sample across the conventional multiple plate condenser without using some form of leads. The errors these introduce, particularly at frequencies above 1 megacycle, may not be neglected where accuracy is required.

RECOMMENDATIONS

Some of the recommendations drawn which have been submitted to the Society for their use in preparing standards are as follows:

METHODS FOR MEASURING DIELECTRIC CONSTANT AND POWER FACTOR OF GLASS

Preparation of Samples:

Cleaning.—Use mild soap and water, rinse with tap or distilled water.

Drying.—Drain to remove visible moisture, then store in desiccator until ready for measurement (not less than 15 min.).

Caution: Do not touch with hands after cleaning.

Application of Electrodes:

In general, methods covered by D 150-36 T[4] are suitable. Care should be exercised to keep samples clean during application of electrodes.

Measurements:

In general, methods covered by D 150-36 T[4] are suitable. The following minor modifications are suggested as being desirable in the case of glass.

1. The bridge method is recommended for frequencies from 60 cycles to about 100 kc.
2. The substitution method is recommended for frequencies from about 100 kc. to about 1 megacycle.
3. If the substitution method is used above about 1 megacycle, greater care must be exercised to avoid difficulties from lead inductances and stray capacitances.
4. It is recommended that the circuit shown in D 150 – 36 T[4] be modified so as to permit the operator to switch from the sample to the standard rather than to connect the sample across the standard.

While the skeleton specification summarizes the agreements reached at the meeting, additional points raised during the discussion may be of interest.

Application of Electrodes:

It was agreed that foil electrodes are satisfactory but that for best results the petrolatum should be in good condition, particularly as regards freedom from excessive moisture content. Considerable pressure should be applied to the foil electrodes to insure a smooth application and to reduce the thickness of petrolatum to a minimum.

The results obtained with these measurements in common with so many others is usually in proportion to the care exercised. For example, the frequency range limits given for bridge measurements are by no means absolute. Likewise, the doubts entertained as to the accuracy of the substitution method above 10 megacycles are lessened as the knowledge and skill of the operator increases.

J. T. Littleton, of the Corning Glass Works, as Chairman of Section B of Subcommittee V of Committee D-9 is largely responsible for organizing the work of this group and keeping it moving. R. C. Dehmel of the Bell Telephone Laboratories is responsible for preparation of the composite curves and accumulating the data turned in by the cooperating laboratories. Data were taken by the following men:

E. M. Guyer of Corning Glass Works
R. C. Dehmel of Bell Telephone Laboratories
L. E. Herborn of Bell Telephone Laboratories
R. N. Bushman of General Electric Co.
G. K. Miller of Purdue University
L. J. Berberich of Westinghouse Electric and Manufacturing Co.
P. A. Richards of RCA Manufacturing Co., Inc.

In addition, valuable material has been added by Miss Louise M. McDowell of Wellesley College, R. F. Field of General Radio Co., J. W. Kennard, J. G. Chaffee, and K. G. Coutlee of Bell Telephone Laboratories, and H. R. Meahl of General Electric Co.

TESTS OF GLUED LAMINATED WOOD BEAMS IMPREGNATED WITH CREOSOTE

By William A. Oliver[1]

Synopsis

The purpose of this paper is to report upon a series of tests of small glued and creosote treated laminated wood beams. The size of the specimens was that recommended by the Society for small clear specimens to be tested in cross bending—2 by 2 in. in cross-section on a 28-in. span loaded at mid-span. Control beams of the same size were also tested. For each treated specimen there was a corresponding plain solid beam and a glued laminated untreated one.

In order to study the effect of the aging of the glue and of the seasoning of the wood, the tests were carried out at intervals over a period of a year. Data on moisture determinations made at the time of the tests and material on density are also included in the report. The effect of moisture content and seasoning upon the strengths obtained are discussed.

The results obtained in these tests indicate that given a proper period of seasoning the same strengths may be expected from glued and creosote treated beams as are expected from plain solid beams or glued untreated ones.

Glued timber construction has become well established both in the United States and abroad and it can be considered as one of our standard methods of construction. However, while laminated glued timber structural units are common enough, these same units impregnated with a preservative material have not been used and as far as the author has been able to ascertain very little is known regarding the effect of preservatives upon the properties of the glue. This paper reports upon a series of tests made to investigate the strength of glued laminated wood beams treated with creosote.

Test Specimens

The test specimens were small beams 2 by 2 in. in cross-section and they were tested on a span of 28 in. with a single load applied at the center line of the beam. It will be noted that this is the size of the A.S.T.M. standard wood beam specified in A.S.T.M. Standard Methods of Testing Small Clear Specimens of Timber (D 143 - 27).[2] As nearly as was practicable the other requirements of that standard were also complied with in order to be able to compare the strengths obtained with similar average strengths which have been reported by the Forest Products Laboratory at Madison, Wis. The testing apparatus which is a modification of the A.S.T.M. standard[2] is shown in Fig. 1.

Twelve specimen groups have been tested with three beams in each group. Each group of three beams was made up and marked as follows: (*a*) a solid plain beam 2 by 2 in. in cross-section, untreated; (*b*) a glued untreated beam made of four $\frac{1}{2}$ by 2-in. laminations; and (*c*) a glued treated beam made of four $\frac{1}{2}$ by 2-in. laminations. The two untreated beams of the group were to act as control specimens for the treated one. The beams were tested with the 2-in. dimension of the laminations horizontal. The specimens were made of Douglas fir of a structural grade.

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[1] Assistant Professor of Civil Engineering, University of Illinois, Urbana, Ill.

[2] 1939 Book of A.S.T.M. Standards, Part II, p. 513.

The beams in each group were cut from the same part of the original stick in order to eliminate as much as possible the effect of annual growth upon variation in strength. In gluing the laminations together, the pieces were brushed clean and the glue was applied on one side only with an ordinary paint brush. Because of the danger of crushing the small pieces, the pressure applied in gluing the specimens was less than that used in gluing full size structural members. A standard casein glue was used.

The treated beams were impregnated with a No. 1, A.W.P.A. creosote after the laminations had been glued together, the treatment being by the Rueping or empty cell process with a retention of 8.33 lb. of creosote per cubic foot of material. Cross-sections cut through the treated specimens showed that the creosote had penetrated throughout the material.

TESTING PROCEDURE

The specimens were tested, three groups at a time over a period of approximately a year so that some information might be obtained regarding the time effect of the creosote upon the strength of the glue. The actual age at the time of testing each series of groups of three was as follows: groups 1, 2, and 3, 16 days; groups 4, 5 and 6, 184 days; groups 7, 8, and 9, 247 days; and groups 10, 11, and 12, 394 days.

On the day of testing, moisture content determinations were made on each plain and untreated beam. Two moisture specimens were cut from each beam subsequent to testing. The determinations were made in an electric oven.

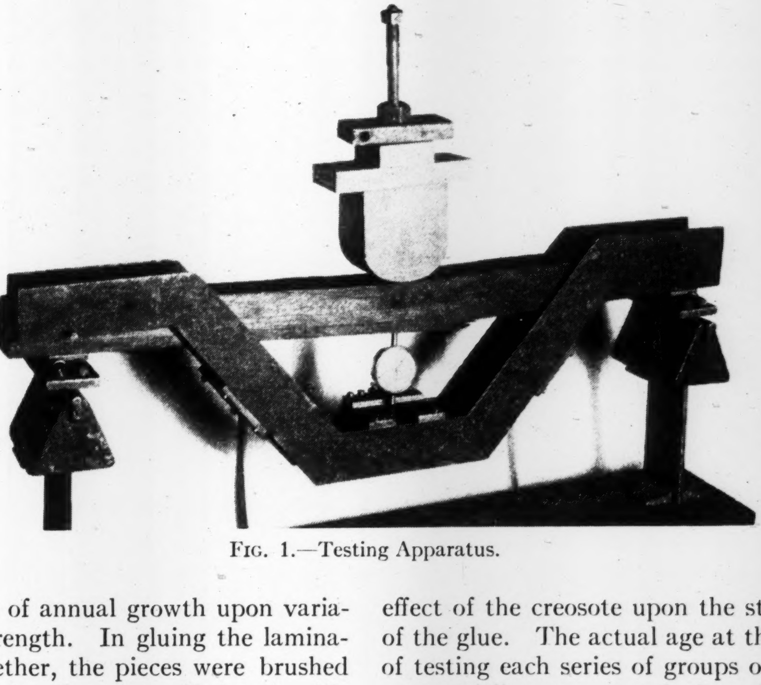

FIG. 1.—Testing Apparatus.

The average moisture content obtained for each beam, based on oven-dry weight, is shown in Table I where the measured depths and widths of the specimens are also shown.

TABLE I.—SIZE AND MOISTURE CONTENT.

Specimen Group[a]	Depth, in.	Width, in.	Average Moisture, per cent
TESTED APRIL 15, 1939. AGE 16 DAYS			
1 (*a*)	1.97	2.01	11.2
(*b*)	1.99	2.01	15.1
(*c*)	2.00	2.05	....
2 (*a*)	1.99	2.00	11.2
(*b*)	2.02	1.99	14.4
(*c*)	2.00	2.05	....
3 (*a*)	1.99	1.98	11.8
(*b*)	1.99	2.00	14.5
(*c*)	2.02	2.05	....
TESTED SEPTEMBER 30, 1939. AGE 184 DAYS			
4 (*a*)	1.98	1.98	10.9
(*b*)	1.97	2.01	13.7
(*c*)	2.03	1.98	....
6 (*a*)	1.97	1.97	10.8
(*b*)	2.00	2.01	12.3
(*c*)	2.04	1.97	....
TESTED DECEMBER 2, 1939. AGE 247 DAYS			
7 (*a*)	1.99	1.97	9.5
(*b*)	1.98	2.00	9.5
(*c*)	1.98	1.98	....
8 (*a*)	1.97	1.98	8.8
(*b*)	1.97	1.94	9.4
(*c*)	1.97	2.00	....
9 (*a*)	1.98	2.02	9.3
(*b*)	1.99	1.99	8.8
(*c*)	2.07	2.02	....
TESTED APRIL 27, 1940. AGE 394 DAYS			
10 (*a*)	1.95	2.00	9.0
(*b*)	1.95	1.97	7.5
(*c*)	1.97	2.01	....
11 (*a*)	1.97	1.99	8.2
(*b*)	1.95	1.97	7.9
(*c*)	1.94	1.97	....
12 (*a*)	1.97	2.00	8.8
(*b*)	1.92	1.94	8.3
(*c*)	1.97	2.00	....

[a] (*a*) = Plain beam; (*b*) = glued, untreated beam; (*c*) = glued, treated beam.

In testing the beams, a deflection reading was taken on an Ames dial (see Fig. 1) at zero load and at 100-lb. increments up to the point where failure was apparently imminent, at which time the dial was removed. The ultimate load from which the modulus of rupture was obtained was also observed. The load was applied through the moving head of a 30,000-lb. Riehle testing machine.

DISCUSSION OF RESULTS

The data obtained from the bending tests are shown plotted on Figs. 2 to 12, inclusive. Each figure represents a single specimen group. Extreme fiber stresses computed by means of the beam formula have been plotted against total deflection. The proportional limit point and sketches showing type of beam failure are also shown on these figures.

TABLE II.—MECHANICAL PROPERTIES.

Specimen Group	Stress at Proportional Limit, psi.	Modulus of Rupture, psi.	Modulus of Elasticity, psi.
TESTED APRIL 15, 1939. AGE 16 DAYS			
1 (*a*)	8 300	13 600	2 076 000
(*b*)	6 400	12 000	2 013 000
(*c*)	6 200	10 200	1 944 000
2 (*a*)	7 400	12 200	2 019 000
(*b*)	6 600	12 300	1 882 000
(*c*)	6 900	10 800	2 189 000
3 (*a*)	8 000	12 200	1 927 000
(*b*)	5 700	11 300	1 740 000
(*c*)	5 500	9 000	1 941 000
TESTED SEPTEMBER 30, 1939. AGE 184 DAYS			
4 (*a*)	7 680	13 500	1 584 000
(*b*)	8 160	15 100	1 708 000
(*c*)	8 800	13 700	1 989 000
6 (*a*)	9 350	13 700	2 189 000
(*b*)	8 530	13 600	2 074 000
(*c*)	9 100	9 600	1 858 000
TESTED DECEMBER 2, 1939. AGE 247 DAYS			
7 (*a*)	9 160	13 700	1 871 000
(*b*)	9 700	14 500	1 914 000
(*c*)	8 840	11 100	2 161 000
8 (*a*)	9 600	15 800	2 056 000
(*b*)	10 220	15 600	2 139 000
(*c*)	11 150	15 200	2 305 000
9 (*a*)	7 980	10 500	1 468 000
(*b*)	7 200	10 100	1 625 000
(*c*)	6 800	11 700	1 774 000
TESTED APRIL 27, 1940. AGE 394 DAYS			
10 (*a*)	9 200	16 700	1 916 000
(*b*)	9 760	16 000	2 094 000
(*c*)	9 360	15 500	2 487 000
11 (*a*)	9 420	15 400	2 139 000
(*b*)	10 100	15 700	2 218 000
(*c*)	9 580	16 800	2 627 000
12 (*a*)	9 660	17 500	2 006 000
(*b*)	9 850	16 900	2 535 000
(*c*)	10 720	16 200	2 504 000

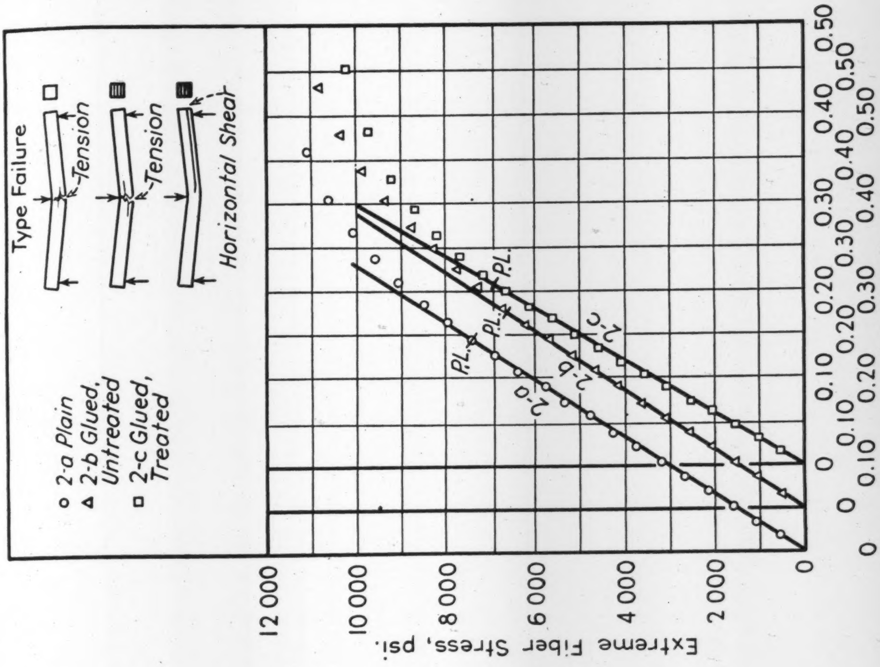

FIG. 3.—Specimen Group 2.

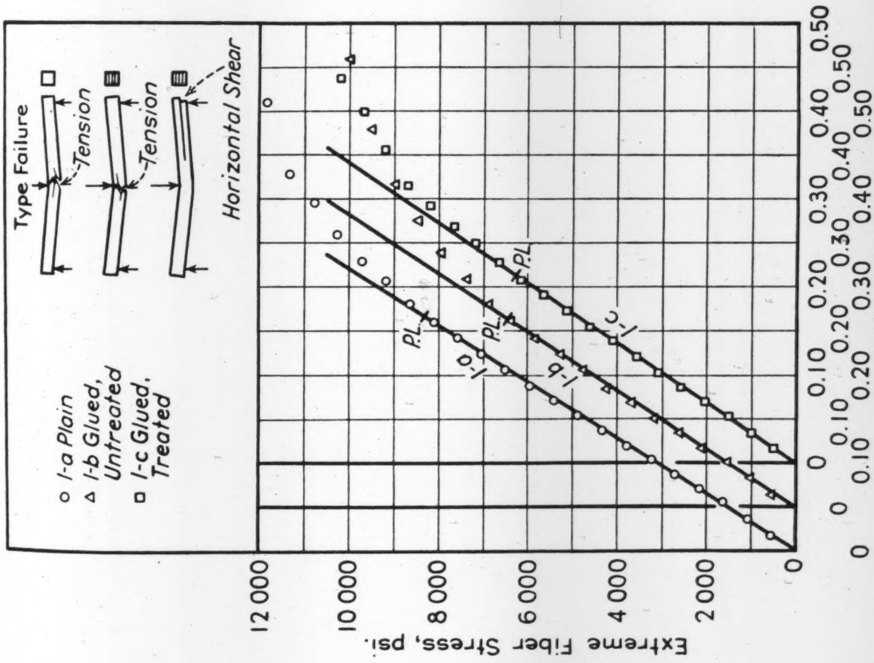

FIG. 2.—Specimen Group 1.

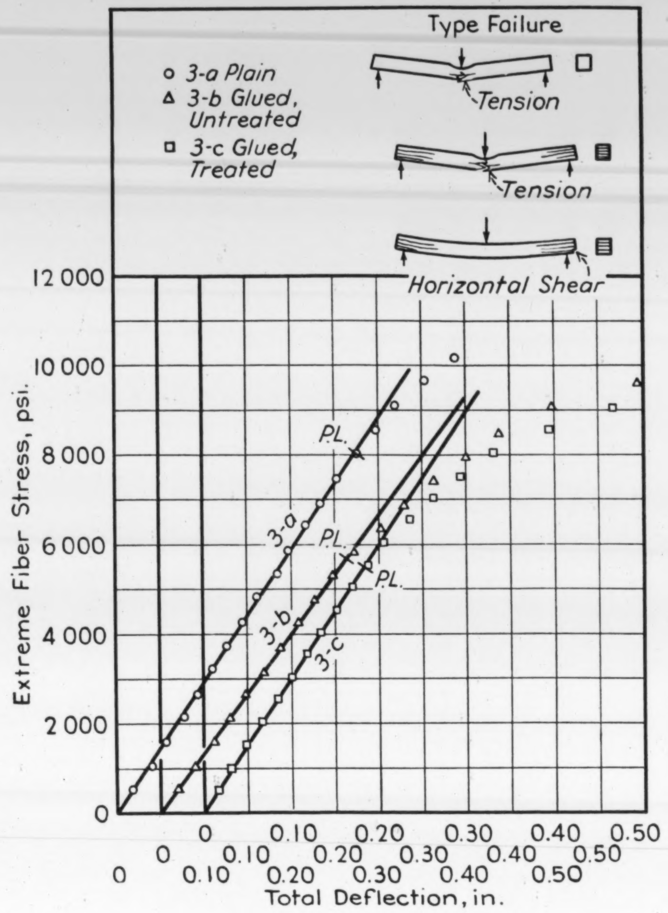

FIG. 4.—Specimen Group 3.

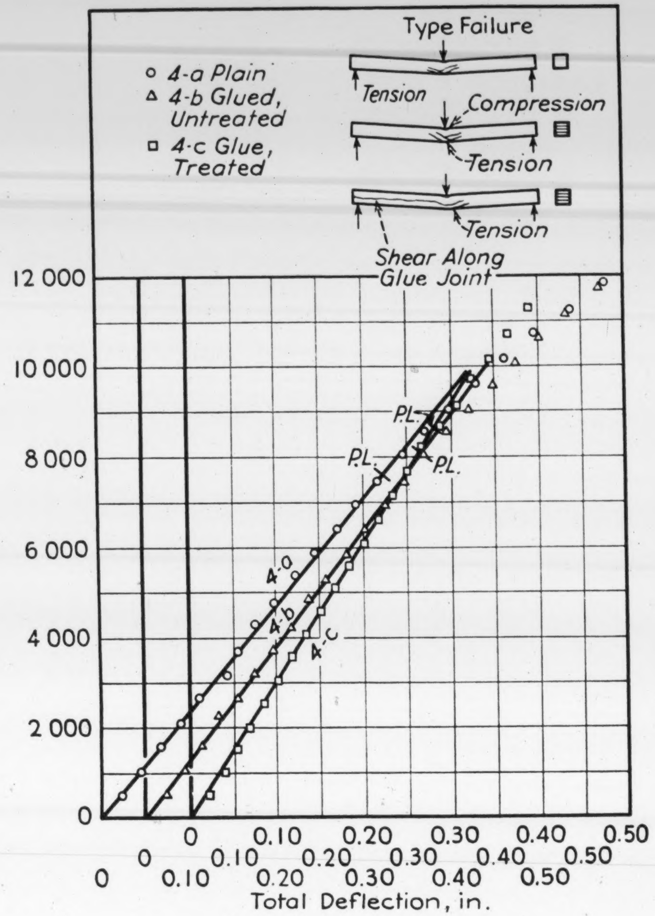

FIG. 5.—Specimen Group 4.

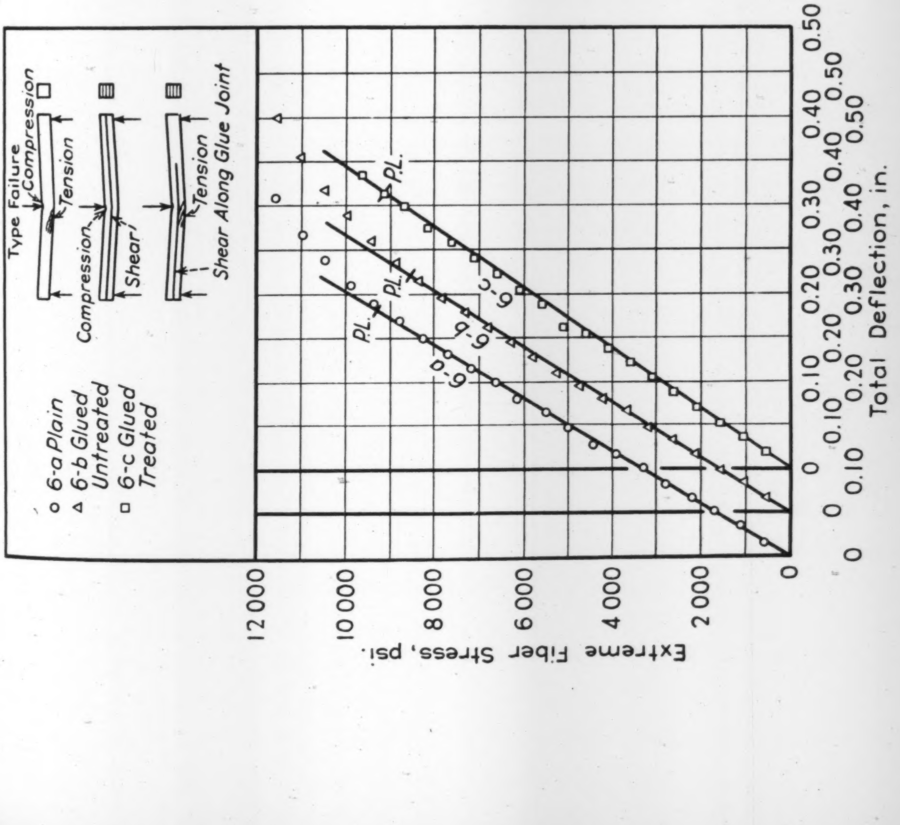

FIG. 6.—Specimen Group 6.

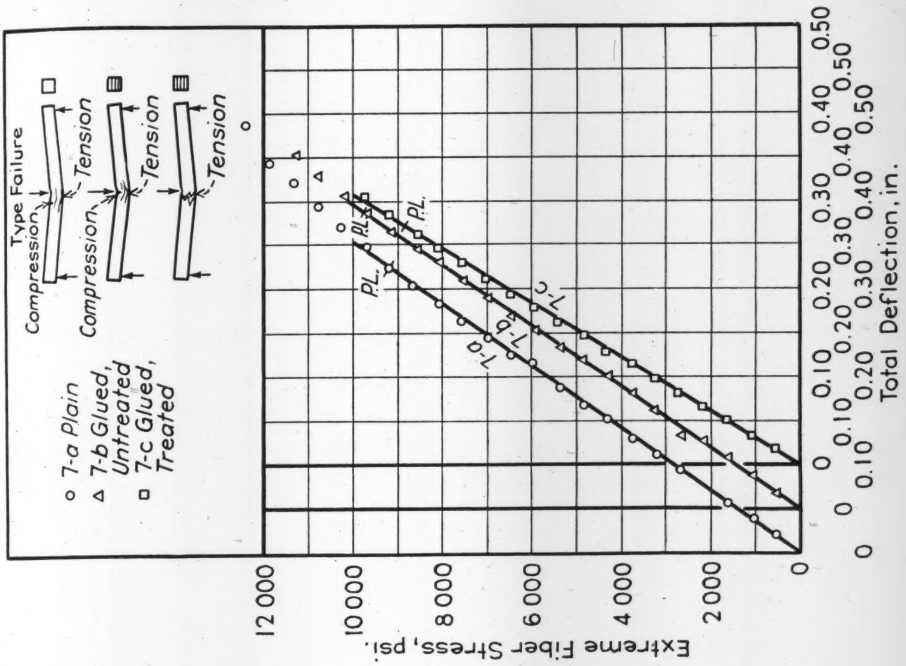

FIG. 7.—Specimen Group 7.

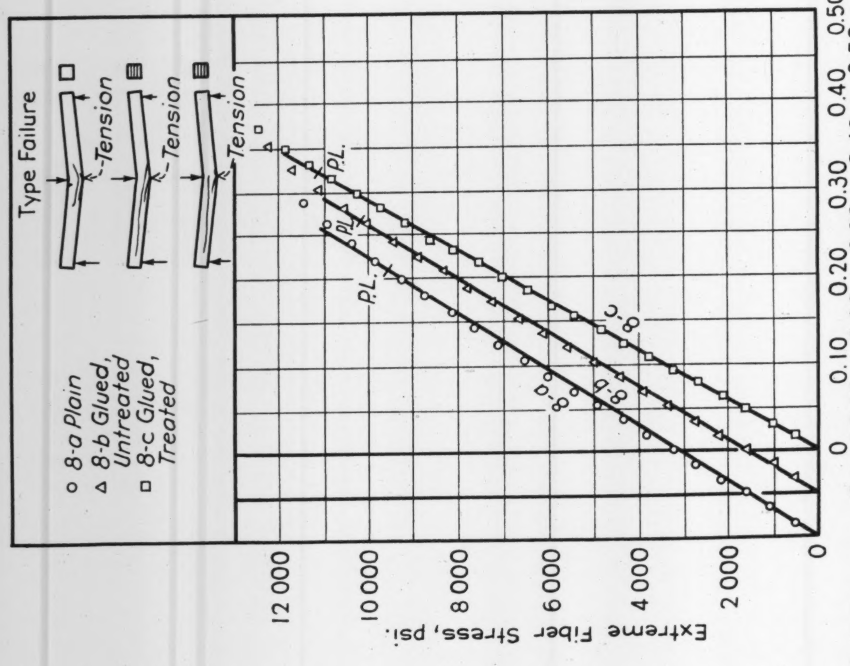

FIG. 8.—Specimen Group 8.

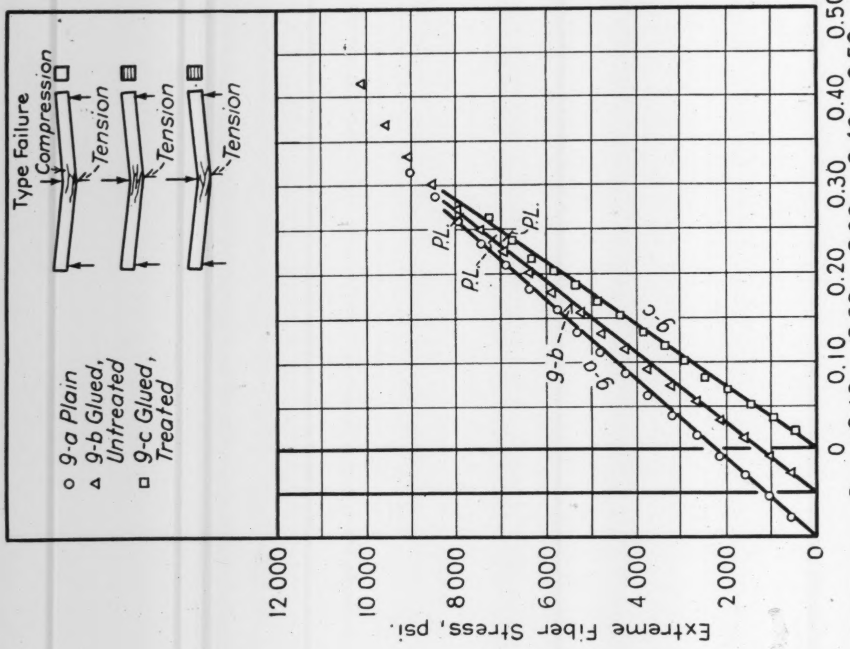

FIG. 9.—Specimen Group 9.

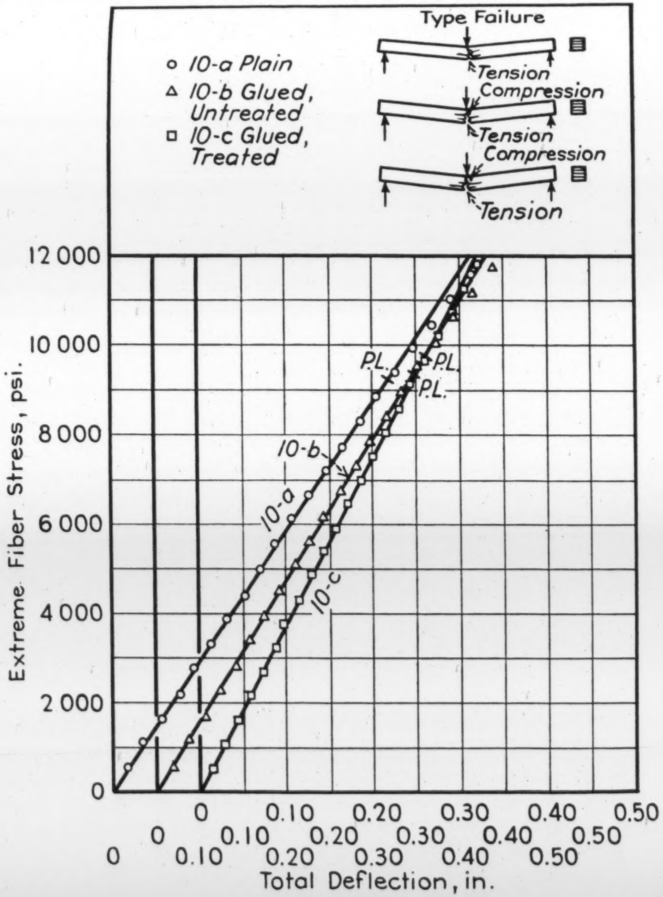

FIG. 10.—Specimen Group 10.

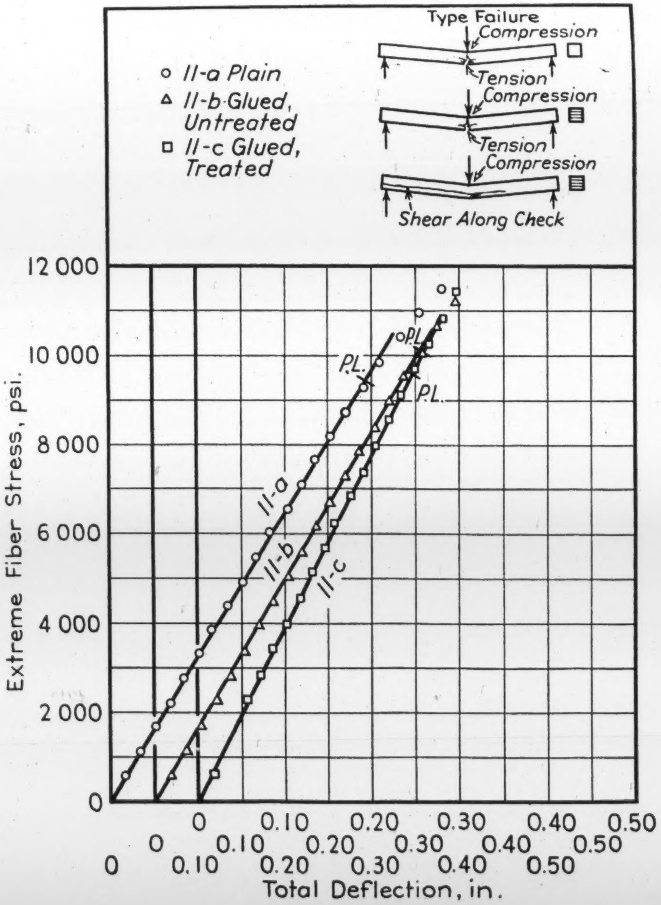

FIG. 11.—Specimen Group 11.

The mechanical properties obtained from the data have been tabulated in Table II. It should be noted here that a blunder was made in testing beam 5(*c*) which made the results of that test worthless. Consequently none of the data from the group 5 specimens are included.

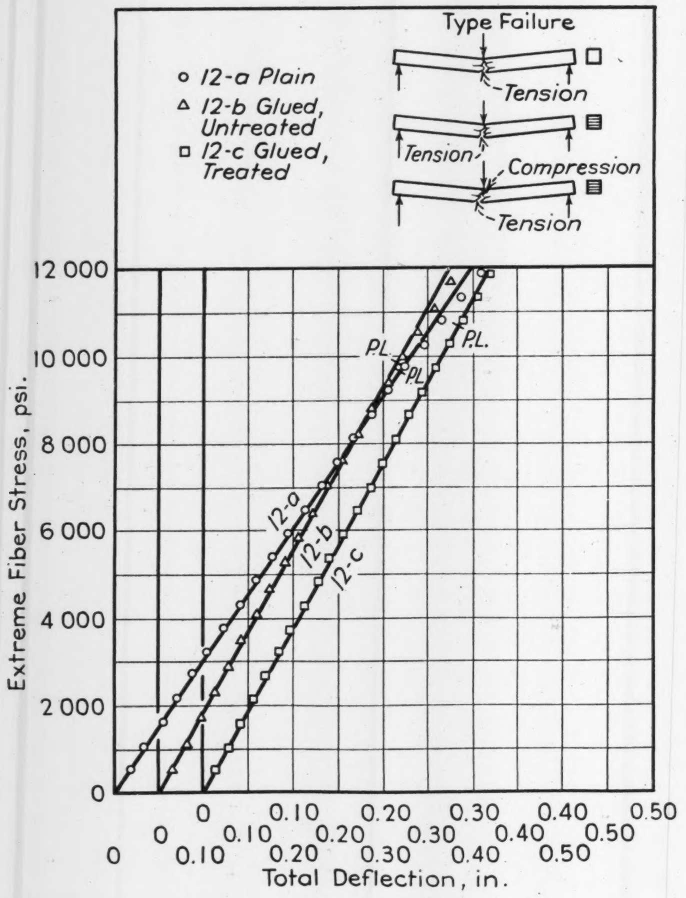

FIG. 12.—Specimen Group 12.

While the fact that the specimens were made of Douglas fir of a structural grade classifies them to a certain extent as far as density is concerned, specific gravity tests of specimens cut from beams 1(*a*), 2(*a*), and 3(*a*) were made giving values of 0.53, 0.52, and 0.49, respectively. These values of specific gravity were obtained in an oven-dry condition. The material of these three specimens was typical of the material used throughout. Cross-sections of the glued and treated laminated beams are shown in Fig. 13 from which an approximate visual determination of density may be made. The darker material in the annual ring is the summer wood. It should be remembered that the beams were 2 by 2 in. in cross-section.

The variation of moisture content with age of specimen is plotted on Fig. 14. As stated above the average values of the moisture content of each beam are given in Table I. The curves of Fig. 14 represent the average of each group at the time of testing. These graphs show a decrease of moisture content with age for all specimens. It will be noted that the moisture content of the glued untreated beams was considerably higher than that of the plain beams in the first two series of tests. This would be due to the moisture imparted by the glue. As time elapsed and the glued material had the opportunity to season, the moisture contents again became equal or nearly so.

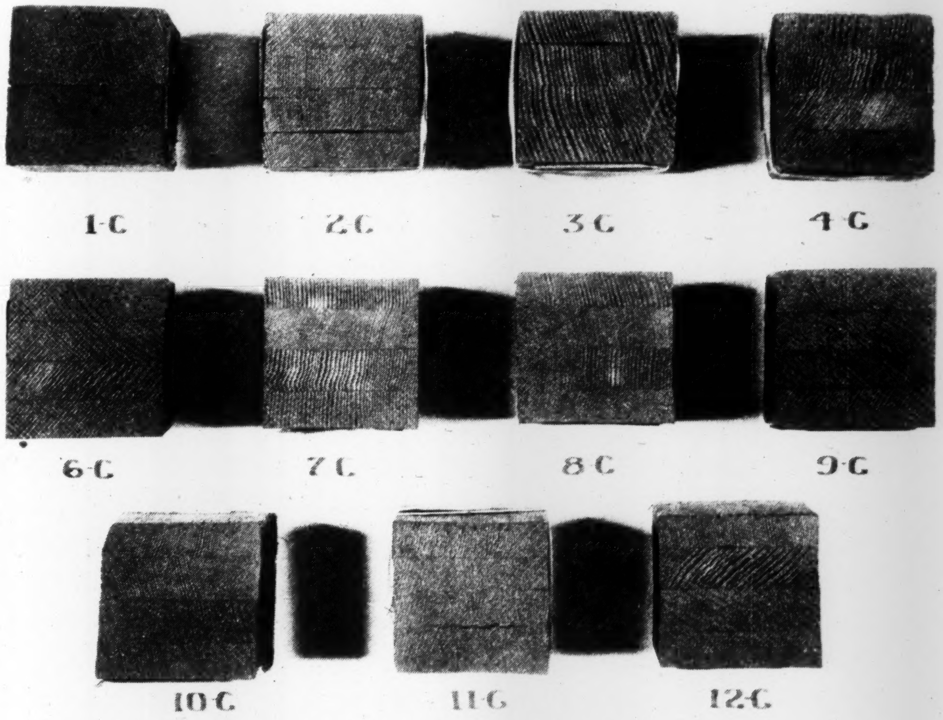

FIG. 13.—Cross-Sections of Glued Treated Beams.

While no moisture content could be obtained quantitatively from the glued treated beams it was observed at the time of testing the first groups that due both to the gluing and to the subsequent creosote treatment these beams had the appearance of being thoroughly soaked. At the time of testing the first groups—age 16 days—the creosote treated specimens were bleeding so freely that they were difficult to handle. During the later tests, however, observation showed that they had, also, become thoroughly seasoned.

The variations of the proportional limits with age are shown in Fig. 15. The values of the proportional limits were obtained from the stress-deflection curves of Figs. 2 to 12 and are shown in Table II. The proportional limit was determined as the point where it was estimated that the stress-deflection curve departed from the tangent.

Two things will be observed from the graphs of Fig. 15. First, the value of the proportional limit tends to increase

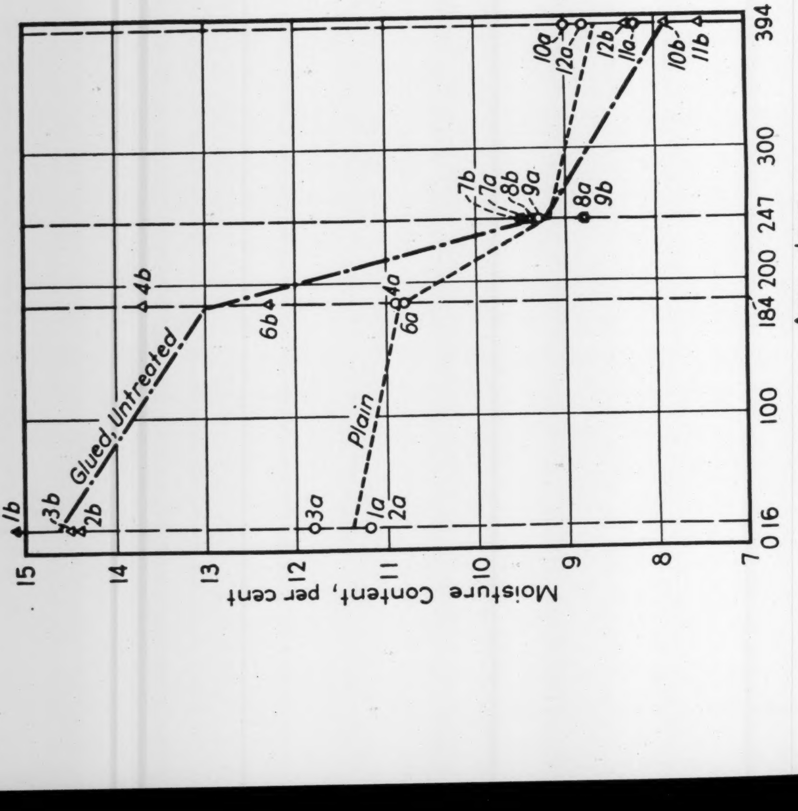

FIG. 14.—Age-Moisture Content Curves.

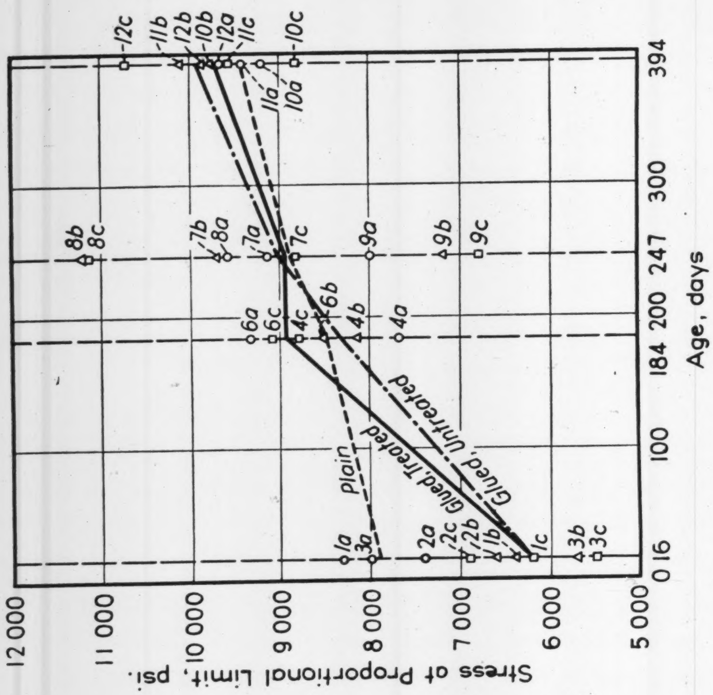

FIG. 15.—Age-Proportional Limit Curves.

with age for all specimens and second, the proportional limits of the glued untreated and the glued treated specimens, while in general lower in the early tests, became equal or greater in value than those of the plain beams in the later tests. The curves are drawn for the average of the tests of a given age. However, the values obtained for the individual beams have been plotted so as to show the variation from the mean. This increase of the proportional limit with age is due primarily to the effect of seasoning.

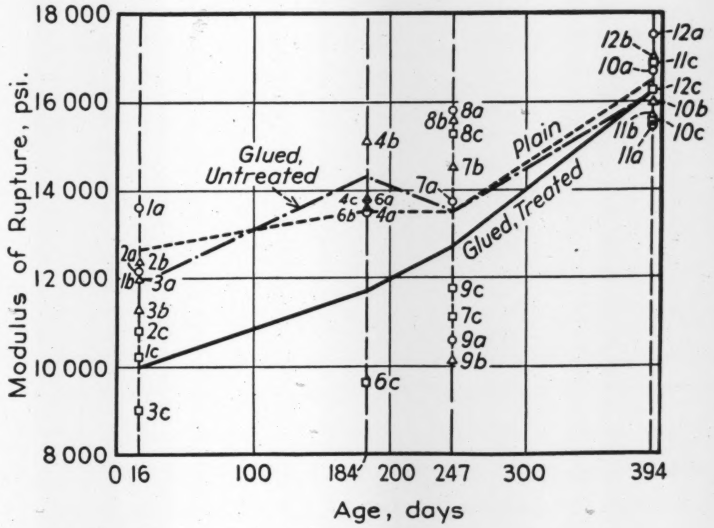

FIG. 16.—Age-Modulus of Rupture Curves.

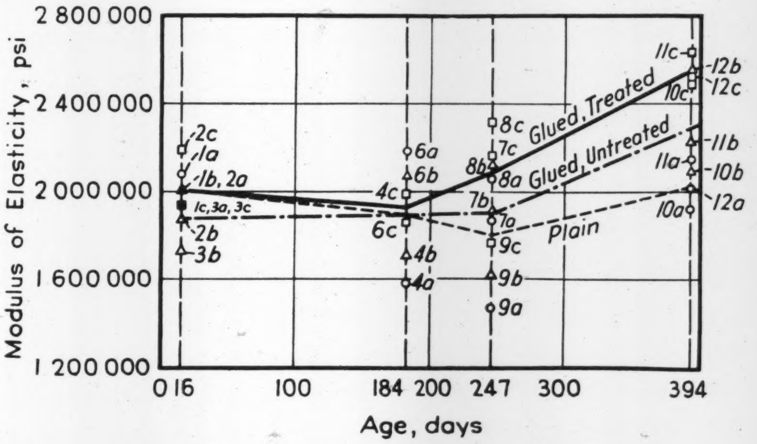

FIG. 17.—Age-Modulus of Elasticity Curves.

The values of the modulus of rupture and the modulus of elasticity obtained from the tests are shown in Table II. The modulus of rupture - age and the modulus of elasticity - age variations are shown in Figs. 16 and 17, respectively. Here again, in the case of both of these properties, as with the proportional limit, the values tend to increase with age. And here again, also, while there was quite a wide range of values obtained for a given age group, due no doubt to errors in testing as well as to neglected variables in the material itself, the very distinct tendency toward increase in strength with age is due mainly to the seasoning of the specimens. In any case it is evident that after proper seasoning both the glued untreated and glued treated beams compare very favorably in strength with the plain ones.

There is some evidence in the data obtained in these tests to indicate that the glued laminated beam is stiffer and stronger than a plain beam of the same dimensions. In a paper on "The Properties of Built-Up Wooden Beams"[3] the author, Peter Landsem, makes the statement that the glued laminated beam by the proper choice of materials may be stiffer and stronger than an equivalent solid beam. Furthermore, the results of a series of tests on glued laminated wood arches carried out at the University of Illinois, showing that a properly glued joint is stronger than the surrounding material, lead to the same conclusion. As stated above the results of these beam tests are further evidence of the truth of this statement.

In the table prepared by the Forest Products Laboratory giving "Strength Properties of Some Commercially Important Woods Grown in the United States," appearing on p. 50 of "Wood Handbook," the mechanical properties of three species of Douglas fir are listed. The values there listed for the three strength properties which have been determined in these tests are as follows for 12 per cent moisture content: proportional limit, 6300, 7400, and 8100 psi.; modulus of rupture, 9600, 11,300, and 11,700 psi.; modulus of elasticity, 1,400,000, 1,610,000, and 1,920,000 psi. A reference to Table II will show that the values obtained for these properties in the tests reported herein are in general within the range of the values established by the Forest Products Laboratory. A more accurate comparison could probably be made by correcting the values shown in Table II to a 12 per cent moisture content.

In the sketches showing the type of failure (see Figs. 2 to 12), any progression of failure which resulted in a break on the lower side of the beam under the load has been designated as a tension failure providing it was not preceded by a horizontal shear failure. The designation, horizontal shear failure, carries the usual significance. The sketches show that twenty-eight specimens out of the eleven groups of three had a tension failure. In the case of specimens 4(*c*) and 6(*c*) this was followed by a shearing separation along a glued joint between laminations.

Five specimens failed in horizontal shear. Specimens 6(*b*) and 11(*c*) failed in horizontal shear but the failure did not occur in a glued joint. Specimens 1 (*c*), 2 (*c*), and 3(*c*), the glued treated beams of the first three groups tested (age 16 days), all failed in horizontal shear along a glued joint. This last statement might appear to be of some significance with regard to the strength of the glued treated beams. However, it should be remembered that these beams when tested had the appearance of being very wet due to the glue and to the creosote, and in the later tests, when the material had had time to season and the glue had had time to set up, this horizontal shear type of failure no longer occurred.

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[3] Group C—Organic Materials, Sub-Group 3, p. 486, London Congress, International Association for Testing Materials, April, 1937.

This predominance of the tension type of failure indicates that the glued treated laminated beams behaved in a manner similar to both the plain beams and the glued untreated beams. Even where the beams failed in horizontal shear the strengths obtained do not compare unfavorably with the strengths of the other beams of the same age.

SUMMARY

The results obtained in this series of tests show that for all practical purposes the glued and treated laminated beams, when properly seasoned, have the same strength as either the plain beams or the glued untreated laminated beams. A full-size glued laminated wood beam may be made up of a number of smaller pieces with a greater number of the natural defects eliminated than is possible with a solid beam of the same size. Consequently, it seems logical to expect that the results obtained from full-size tests would be even more favorable towards the glued treated beams.

Acknowledgments:

The tests were made in the Civil Engineering Laboratories of the Engineering Experiment Station of the University of Illinois. Dean M. L. Enger is Director of the Experiment Station and Prof. W. C. Huntington is Head of the Department of Civil Engineering. McKeown Brothers Co. of Chicago made the specimens. The material was furnished by Edward Hines Lumber Co., Chicago, and approved for testing by Mr. Kramer of the Chicago Lumber Institute. The beams were creosote treated by the Joslyn Manufacturing and Supply Co., Franklin Park, Ill.

DISCUSSION

MR. L. J. MARKWARDT[1] (*by letter*).—Mr. Oliver's study on treated glued laminated beams provides specific data in a field that has so far received but limited attention.

Studies by Wilson[2] at the Forest Products Laboratory, have shown the advantages and possibilities of laminated construction, both for straight and curved members. Most practical of the glues for laminated construction has been casein, which has long working life and does not require heat. Hence, casein glue has been used for the laminated arches and beams in the many buildings employing laminated construction that have already been erected. Since casein glued joints are water resistant but are not waterproof to the same degree as those with the newer synthetic resins, the applications have been limited largely to inside dry locations. An entirely new field of use for laminated construction would be possible if and when technique is developed to permit the setting of synthetic resins in large structural members, and if laminated material can be treated with wood preservatives to afford decay resistance for use under exposed conditions.

There are apparently but few data available on the effect of creosote on the strength of glue joints. Tests reported by Brouse[3] show that the shearing strength of the joints of thin plywood panels made up with casein glues are not markedly affected by creosote impregnation subsequent to the gluing of the plywood. Indeed, under severe moisture exposure conditions, such treated panels gave much better performances than the untreated panels otherwise similarly constructed. This would indicate that casein glue and creosote are not incompatible, and that at least over short periods glued joints retain their integrity in the presence of creosote.

In his study Mr. Oliver also used casein glue in the laminated beams that were subsequently treated and tested. In connection with the test results, it should be noted that the Standard A.S.T.M. static bending specimen[4] was used. These specimens have a 14:1 span-depth ratio, and experience has shown that horizontal shear failures with normal wood of all species are very uncommon. The fact that 5 of the 33 specimens failed first in horizontal shear must accordingly be regarded as indicating some significant weakening effect. This would suggest that certain factors in the glue, gluing operations, seasoning of the glued joints prior to treatment, or the impregnation process might be the predisposing cause of the shear failures reported. Considering these possibilities in the light of the fact that 3 of the 5 horizontal shear failures occurred in the first three groups tested (age 16 days) and that the horizontal shear failures in the other two instances were in partially seasoned or seasoned specimens and did not occur along the glue line, the implication of a permanent weakening, possibly as a result of the temperatures and pressures employed in treating process, cannot be escaped. Whatever the cause, these shear failures, although associated with relatively high loads, have implications which should not be ignored.

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[1] Chief, Division of Timber Mechanics, Forest Products Laboratory, Madison, Wis.

[2] T. R. C. Wilson, "The Glued Laminated Wooden Arch," *Technical Bulletin 691*, U. S. Department of Agriculture, October, 1939.

[3] Don Brouse, "Methods of Increasing Durability of Plywood," *Woodworking Industries*, February, 1932, pp. 30, 31, 33.

[4] Standard Methods of Testing Small Clear Specimens of Timber (D 143 - 27), 1939 Book of A.S.T.M. Standards, Part II, p. 513.

Extensive studies on the effect of treating methods on wood (not laminated) have shown that the size of the beam has an important influence on the internal stresses set up during the seasoning process. In large beams, also, there is a greater tendency to check than in smaller ones, and with checks in beams there is a concentration of stress at the bottom of the check which often promotes failure. Mr. Oliver properly does not conclude that the results of the study on the small treated laminated beams would be applicable to large size members. The importance of avoiding such a conclusion cannot be emphasized too strongly because of the known inter-effect of size and treatment method on strength.

The fact that the decrease in moisture content of the beams, rather than the increase in age, was primarily responsible for the increase in the mechanical properties as shown in Figs. 15, 16, and 17 may perhaps have been underemphasized. It is pointed out rightly that the tendency toward increase in strength with age is due mainly to the seasoning of the specimens. This should be kept in mind in connection with the analysis of Figs. 14 to 17, inclusive, for there is, of course, no reason to expect the large increase in properties with age which is shown if no moisture change occurred.

Mr. Oliver effectively points out the possibility of making up full-sized glued laminated wood beams from small, relatively clear pieces, thereby obtaining a large structural member free of natural defects and hence of higher strength than a solid wood member containing defects. It is interesting to point out also in this connection the possibility in a laminated beam or arch of using relatively clear wood for the outer highly stressed laminations, and lower grade material containing knots for the inner laminations. Wilson[2] has shown that up to 60 per cent of low-grade material can be used in the inner laminations without materially affecting the strength. It is interesting to note also that short lengths can be employed by the simple expedient of glued scarf joints, which are not difficult to fabricate in the shop.

Mr. Oliver is to be complimented on his study in this field. It is hoped that this work can later be extended to include larger beams and variable span-depth ratios. Then looking forward to still greater utilization possibilities, the development of large, laminated, structural units for exterior use, bonded with a waterproof synthetic resin glue instead of casein and treated with a wood preservative to provide decay resistance, would greatly widen the field of wood constructions for service under exposed conditions.

MR. WILLIAM A. OLIVER (*author's closure by letter*).[5]—The author wishes to express his appreciation to Mr. L. J. Markwardt for his very interesting discussion. He considers that the points which Mr. Markwardt has brought out and emphasized have added greatly to the value of the original paper.

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THE EFFECT OF RATE OF LOADING ON TENSILE STRENGTH OF CORD AND YARN

By M. Castricum[1] and A. N. Benson[1]

Synopsis

Test results obtained on an inclined plane cord testing machine, having a variable speed drive, have been analyzed to show that there is a linear relation between strength and the logarithm of the rate of loading. The equation obtained is also shown to apply to previously published data on rayon yarns. It is also shown that this equation may be used in the range of extremely low rates of loading and it has been applied to earlier data published on the strength of cotton ropes under sustained loads.

The equation relating the rate of loading and strength contains an experimental constant, *K*. A table is included giving the value of this constant for a number of samples.

It has long been recognized that the pendulum type of cord tester introduces an extraneous factor into test data, in that the rate of loading is not constant. Haven[2] proposed a new type of machine, the inclined plane serigraph, which would give a uniform rate of loading. Hunter[3] showed that the rate of loading would still be a factor unless a standard rate, based on grams per denier per minute, were used. Tests reported by Scroggie and Castricum[4] verified the fact that results obtained on the inclined plane machine were influenced by rate of loading.

In a study on the effect of rate of loading on rayon yarn, Bellinson[5] comes to the conclusion that there is a linear relation between strength and rate of loading. An examination of experimental data obtained over a wide range indicates that the relation between strength and rate of loading can be better represented by a transcendental equation.

Apparatus

There are two convenient methods for obtaining a variable rate of loading on the inclined plane machine: (1) the carriage may be constructed with variable weights; and (2) a variable speed driving motor may be used. The latter method has the advantage of giving a continuous range of values within the limits of the machine. This makes possible the testing of any size material at exactly a fixed number of grams per denier per minute. The first method has a disadvantage in that the carriage friction tends to change with the weight of the carriage.

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[1] Manager, Textile Division, and Physicist, respectively, Automotive Development Dept., U. S. Rubber Co., Detroit, Mich.

[2] G. B. Haven, "A Constant Load Rate Testing Machine for Textiles," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 653 (1923).

[3] A. S. Hunter, "Importance of Uniformity in the Constant Rate of Loading with the Scott Inclined Plane Serigraph," *Rayon Textile Monthly*, November, 1936.

[4] A. G. Scroggie and M. Castricum, "Inclined Plane Tests on Cotton Yarn," *Rayon Textile Monthly*, February, 1937.

[5] H. R. Bellinson, "Viscose Rayon: Stress-Strain Properties," *Textile Research*, June, 1940, pp. 316-322.

RESULTS

Table I shows some results obtained in the U. S. Rubber Co. Laboratories on a 4/2 tire cord. Each value represents the average of three 25 break tests. The rate of loading is expressed in terms of grams per denier per minute.

The same data are shown in Fig. 1.

These results may be represented by the following equation:

$$S = S_1 + KS_1 \log R$$

where S = strength at a rate of loading of R g. per denier per min.,

S_1 = strength at a rate of loading of 1 g. per denier per min., and

K = an experimental constant.

The regression equation has been determined from the above data by the method of least squares.

$$S = 15.81 + 1.44 \log R$$

$$K = 9.1 \text{ per cent}$$

Table II shows some of the data reported by Bellinson on 150 denier American Viscose rayon. From an analysis of these data Bellinson obtained the following equation:

$$S_2 = 250.76 + 2.22\, R_x$$

where S_2 = strength at a rate of loading of R_x g. per sec.

The same data were analyzed by the authors to give the following equation:

$$S = 249 + 0.164 \times 249 \log R$$

Table II also shows how well the data agree with each equation. Some extrapolated values are included.

In order to determine which equation fits the data best, the coefficients of

TABLE I.—RESULTS ON 4/2 TIRE CORD.

Rate of Loading, g. per denier per min.	Logarithm of Rate of Loading	Strength, lb.
1.25	0.097	15.98
1.74	0.240	16.12
2.43	0.386	16.41
3.48	0.542	16.63
5.55	0.744	16.83
8.70	0.940	17.25
12.50	1.097	17.46
17.35	1.238	17.52
25.00	1.398	17.75

TABLE II.—DATA ON 150 DENIER AMERICAN VISCOSE RAYON.

Rate of Loading		Calculated Strength, g.		Actual Strength, Bellinson Data, g.
g. per sec.	g. per denier per min.	Benson-Castricum Equation	Bellinson Equation	
4.06	1.62	258	260	261.3
5.14	2.06	262	262	260.2
6.04	2.42	266	264	262.4
7.07	2.83	267	266	266.1
8.15	3.26	270	269	270.2
9.36	3.74	272	272	274.7
10.57	4.23	275	274	274.5
12.06	4.82	277	278	273.9
14.17	5.67	280	282	283.1
	0.001	126	251	
	1000	372	5800	

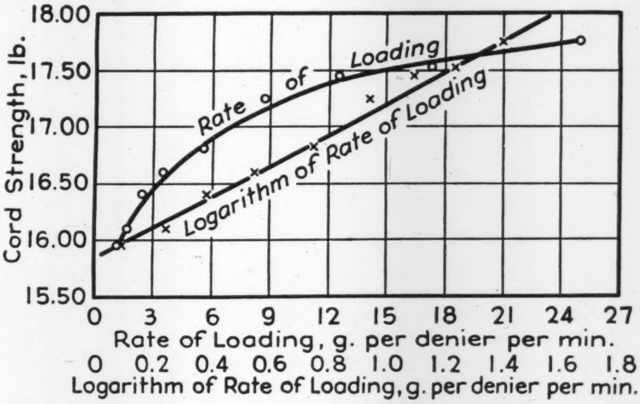

FIG. 1.—Tire Cord Strength *versus* Rate of Loading.

correlation, r^2, between strength and R and then between strength and log R have been calculated. The following values were obtained:

$$r^2 \text{ (strength and } R) = 0.93$$

$$r^2 \text{ (strength and log } R) = 0.89$$

$$\left(r = \frac{\Sigma d_1 d_2}{\eta \sigma_1 \sigma_2} \right)$$

The fact that little difference is found between the coefficients of correlation indicates that within the range of these test results both equations fit the data equally well.

The big difference between the two equations occurs when a wide range of rates of loading are considered. If the 150 denier yarn is subjected to a sudden jerk (1000 g. per denier per min.) the one equation indicates it would have an apparent strength of 372 g. while the other equation says it would have a strength of 5800 g. On the other hand if the yarn is loaded so slowly that a day is required for the break (0.001 g. per denier per min.) then the one equation gives a strength of 126 g. while the other equation gives a strength of 251 g.

TABLE III.—RESULTS ON 15-6 COTTON ROPE.

Breaking Time, hr.	Load, per cent of breaking strength
Regular test—time not given	100
0.12	84
1.4	78
47	69
175	65
3500	49

At the one extreme—low rates of loading—there are some experimental data available in a paper by Lutts and Himmelfarb.[6] In this paper it is shown that ropes loaded at less than their tested strength will nevertheless break after the elapse of sufficient time. The values given in Table III are taken from their results on 15-6 cotton rope.

The data given in Tables I and II are based on an experimental procedure by which the time element becomes the independent variable and the breaking load the dependent variable, whereas the data given in Table III are based on a procedure whereby the load becomes the independent variable and the time element the dependent variable.

The breaking strength value, indicated as 100 per cent in Table III, was obtained by a different procedure from that used for the balance of the tests. Moisture, length of samples, type of jaw, etc., may therefore have introduced some variation. The value of K (from the equation above) and also the correlation between log time and strength have been calculated from data obtained on two types of cotton rope. The following values were obtained:

	r^2	K
15-6 Cotton rope	0.964	7.4
18-6 Cotton rope	0.968	6.9

SUMMARY

The strength of textile materials is effected by the rate of loading. The ralation between the two can be expressed by a logarithmic equation involving one experimental constant. This constant K represents the percentage increase in strength when the rate of loading is increased by a factor of ten. Table IV contains the values of K which have been calculated from the data available at the present time. The indications are that K varies somewhat with the size or strength of the sample under test.

TABLE IV.—VALUES OF K.

Material	K, per cent	Source of Data
150 denier Am. Viscose rayon	16.4	Bellinson
275 denier Cordura rayon	12.2	U. S. Rubber Co.
1100 denier Cordura	9.4	U. S. Rubber Co.
12.9/4/2 American cotton cord	9.1	U. S. Rubber Co.
23/5/3 Sak cotton cord	9.4	U. S. Rubber Co.
15-6 Cotton rope	7.4	Lutts and Himmelfarb
18-6 Cotton rope	6.9	Lutts and Himmelfarb

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[6] Carlton G. Lutts and David Himmelfarb, "The Creep Phenomenon in Ropes and Cords," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1251 (1940).

A METHOD FOR THE TESTING OF ADHESIVE TAPE

By William C. Geer[1] and W. Burton Wescott[2]

Synopsis

In order to determine the adhesion between the surfaces of two bodies one of which, at least, is thin and flexible, the simplest test is to measure the force in stripping one from the other. To attain accuracy, certain requirements must be met.

These requirements were examined in the laboratory of the senior author, as an outcome of which an apparatus was designed and certain precise manipulative methods were developed.

The specific materials tested under these procedures were cloth-backed industrial tape and paper-backed masking tape. The adhesives were pressure-sensitive and contained rubber as the basic ingredient. This paper also describes the methods of preparation and handling of test specimens to avoid personal errors.

A new linear constant-speed rolling apparatus is described by means of which strips of tape may be applied to any desired relatively thin flat surface under conditions of uniformity of pressure and time. A new apparatus for stripping tests includes a mechanism by means of which the force of separation between the tape and the surface is autographically recorded upon a chart.

Chart records show the effects upon adhesion between a standard masking tape and a standard lacquered metal strip of several variants as follows: Thickness of adhesive, variation of load during rolling, rolling by hand as compared with rolling by machine, and effect of load irregularities during rolling.

The results indicate that dependable and reproducible data can be obtained.

In the course of a study in the laboratory of the senior author, at Ithaca, N. Y., of pressure-sensitive adhesives, the authors became convinced that none of the generally accepted methods for the testing of insulating tapes was accurate enough to be a reliable measure of the quality of masking tapes. The appended references are to those more recently published articles which describe methods in current use for the testing of rubber tape and are included here merely for the sake of completeness.

In our studies, the very irregular results which we found seemed to reside partly in how the test specimens were prepared and partly in the apparatus used. After a long examination into the requirements, it became clear that accuracy could be obtained only by the use of apparatus which was designed specifically for the purpose of these tests and by the application of precise manipulative methods.

The purpose of this paper is to present

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[2] Consulting Chemist, Dover, Mass.

a brief description of the apparatus and the methods used, in the hope that it may be of interest not only to those who test masking tape, but also in the field of testing of rubber insulating tapes.

The specific materials tested under these procedures were cloth-backed industrial adhesive tape and paper-backed masking tape. They were received in rolls 1 in. wide and 50 ft. in length. The adhesives tested were pressure sensitive ones containing rubber as the basic ingredient.

The room in which the tests were performed was small and, by means of an air-conditioning unit designed for this room, the temperature was maintained at 70 ± 2 F. throughout the 2 yr. of the study. The humidity was also controlled at 55 ± 5 per cent.

MATERIALS USED

For certain of the tests strips of sheet steel, 2 by 8 by 0.019 in., were used. Each metal strip was gaged with a micrometer in six places and any which varied more than 0.001 in. were rejected. They were then lacquered with Duco lacquer and polished by a professional finisher of automobile bodies. The lacquered strips were cleaned by swabbing with 1-in. squares of surgical cotton dipped in chemically pure carbon tetrachloride. The cotton was manipulated with forceps, so that at no time was it touched by hand. The metal strips at all times were handled by the edges. After cleaning, they were lightly brushed with a clean piece of bleached muslin to remove fibers left from the cotton. No cotton or muslin pieces were reused.

To set up these metal strips for application of the tape, two of them, held by the edges, were placed on the ruled board of the tape cutter, Fig. 2, and marked with red pencil along one side of the strip at 1, 2, and 6 in. from the right hand end. They were then set in position on the surface plate, *H*, of Fig. 1. At each end of these strips, fastened to the surface with tape, was a blank half strip of lacquered metal to maintain a constant level for the roller throughout its journey across the surface plate. The rolling machine was thus made ready for the tape. This machine is described later.

To prepare the tape samples, a roll of tape was placed on the spool of the tape cutter, Fig. 2, so that the tape could be unrolled with the backing side down. Six feet of tape were cut from the roll and rejected. The balance of the roll was then ready for immediate use. Mounting blocks, shown in Figs. 2 and 3 are provided to insure attachment of tabs without contamination of the tape samples.

A paper tab, 1 by $7\frac{1}{4}$ in., provided with a strip of aluminum 1 by $\frac{3}{8}$ in. pierced by a $\frac{3}{16}$-in. hole fastened at one end of the paper tab by means of a small piece of industrial tape, was set on the mounting block. A 13-in. strip of tape was cut from the roll, using the tape cutter, Fig. 2, the tape being removed from the roll at the rate of 6 in. per sec. It is important to observe this exact rate. The test piece of tape was picked up by the ends, turned adhesive side down, held free from any contacts until the 4 in. of tape at the right end of the strip were fastened to the paper tab. The specimen with attached tab was removed to the surface plate and 1 in. at the left end of the tape was fastened to one of the blank metal strips, across the division, and to the left end of the corresponding test metal strip. By means of a hook and cord passed over a pulley, a 100-g. weight was fastened to the tab at the right end of the tape, see Fig. 3. The samples were then ready for rolling into contact with the lacquered metal.

ROLLING MACHINE

It was found by long and careful experiment, that the measured adhesion shown by a given roll of tape varied greatly when the tape specimen was applied to the metal strip by other than precise methods. The purpose of the rolling machine was to roll down the tape samples upon the lacquered metal strips uniformly so that each tape sample was applied to its particular strip under exactly duplicated conditions as to time and pressure.

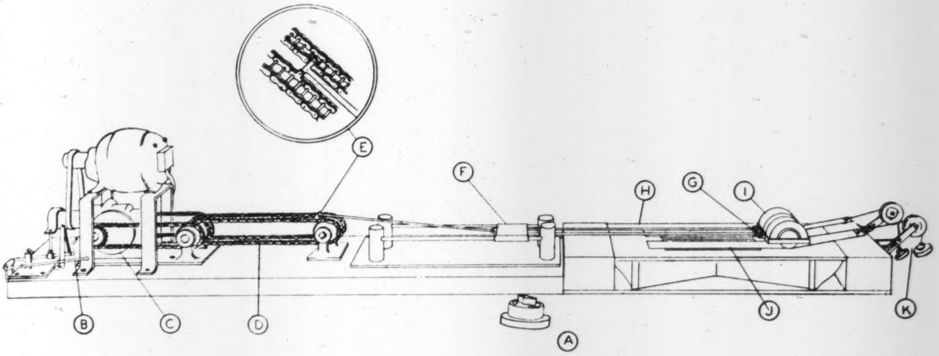

FIG. 1.—Rolling Machine.

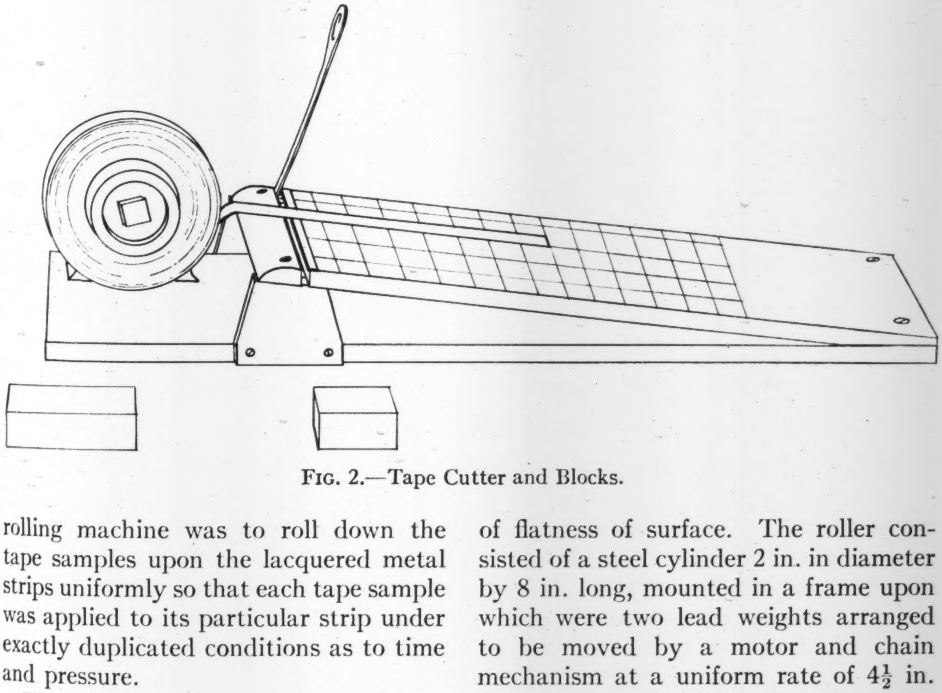

FIG. 2.—Tape Cutter and Blocks.

The rolling machine consisted of three basic parts: (1) a surface plate, (2) the roller with its frame and weights, and (3) the chain drive for the roller. The surface plate had the usual accuracy of flatness to be expected from such an article. This was tested by means of a spherometer, and the one used was found to conform to high accurate uniformity of flatness of surface. The roller consisted of a steel cylinder 2 in. in diameter by 8 in. long, mounted in a frame upon which were two lead weights arranged to be moved by a motor and chain mechanism at a uniform rate of $4\frac{1}{2}$ in. per sec. to and fro across the 9 by 14-in. surface plate. The roller and lead weights together gave a load of $8\frac{1}{2}$ lb. per inch width of tape. The roller was turned, ground, and polished with an accuracy equal to that of the surface plate. It was then chromium plated and regaged.

Reference to Fig. 1 will show the relations of the several parts of the rolling machine. (*A*) is the main switch (*H*) the surface plate, and (*I*) the roller with frame and weights. It will be observed that two test specimens are rolled simultaneously, although one may be a dummy. This is necessary in the interest of uniformity because no scheme of rolling a tape as narrow as 1 in. wide will give a uniform distribution of weight. Even with rolling two at once, a necessary feature of the mechanism is a two-directional joint *G* which permits the roller to move over irregularities in a direction perpendicular to the direction of travel of the roller and, also, in a direction at an angle to the plane of the surface plate. The necessity for this is that tape is highly accurate as to thickness. The parallel rods and carrier *F* insure exactness of the horizontal direction of motion of the roller. Because uniformity of motion over the tape being rolled was found necessary due to the influence upon the adhesion of any dwell upon any given portion of the tape, a chain drive, *D*, for the roller mechanism was developed. *E* shows the attachment of the connecting rod to the chain; *B* is a brake which is necessary to stop the roller at a specific place; *C* is the speed reducer for the motor.

It is to be noted, from the arrangement of the test plates, tape weights, and pulleys, *J* and *K*, that the adhesive upon the tape at no time came in contact with the metal to which it was to be applied, until brought in contact with it by the motion of the roller. This is important. Figure 3 shows this condition more in detail. This shows the surface plate, roller, metal strips, and tape in position; also the blocks diagrammatically shown in Fig. 2 which are used to prevent contamination of the adhesive when applying the tape specimen to the tabs.

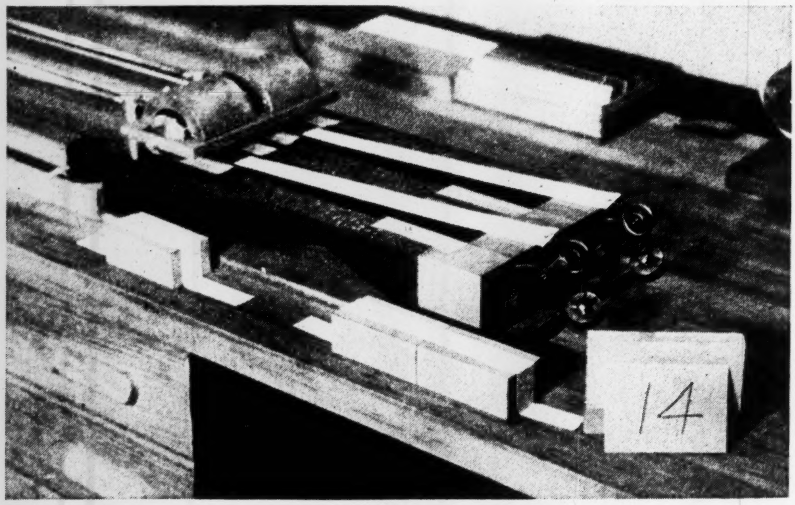

FIG. 3.—Roller Ready for Use.

Two 13-in. strips of tape are cut, mounted on the tabs, and fastened in the outer position on the rolling machine

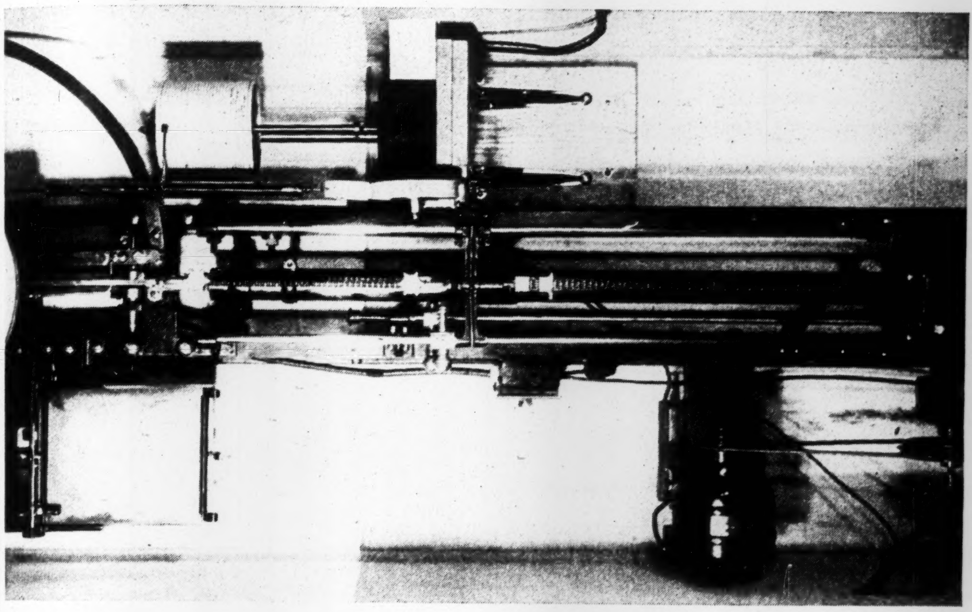

FIG. 5.—Full-Roll Test Adaptation to Scott Machine.

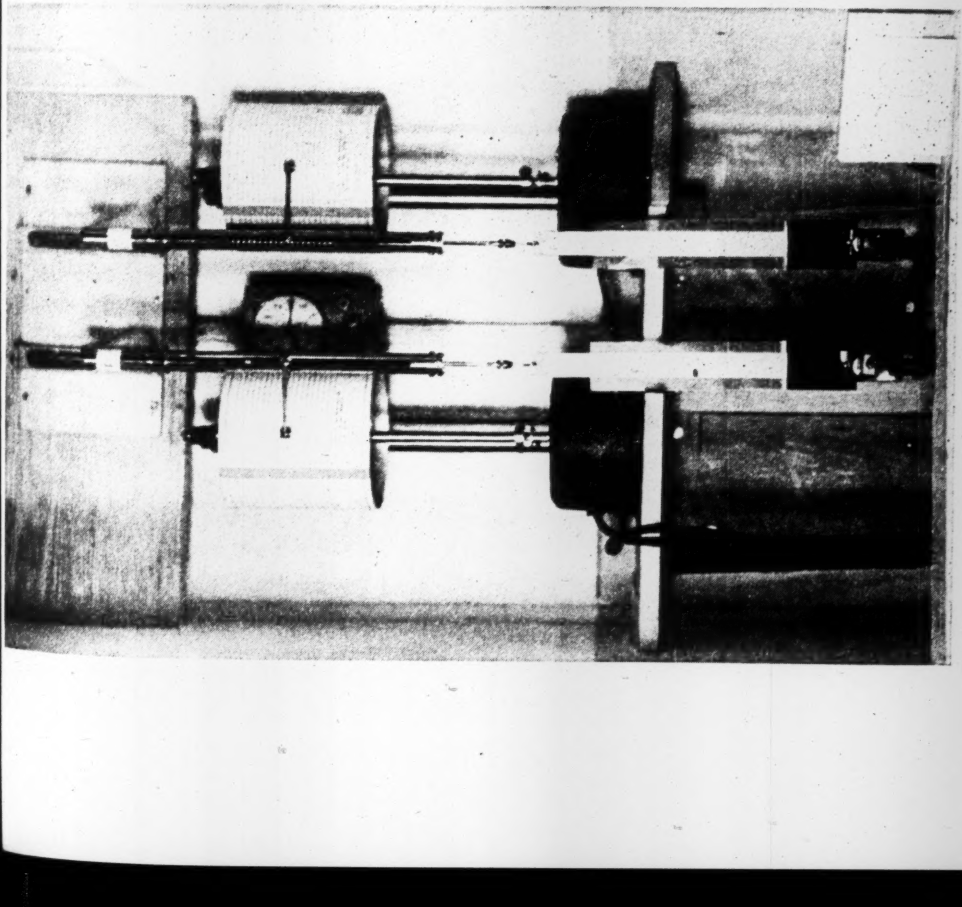

FIG. 4.—Push-Pull Spring Balance.

in the manner already described. The two test pieces of tape, when ready to be rolled down on the lacquered metal strips, are set up as in Fig. 3. The roller passes lengthwise over the tape a total of six times, three times in one direction and three times in the opposite direction. By means of the brake *B*, Fig. 1, the roller can be stopped in position off the metal strips at the left.

When the rolling is finished, the weights are unhooked from the tabs, and the tape and metal strips removed from the surface plate. The $\frac{1}{2}$ in. of tape at the left which overlapped the blank metal strip is folded back and fastened to the underside of the lacquered metal strip. The strips are then set aside to stand for 15 min. before testing.

THE TESTING MACHINE

The stripping test machine is shown in Fig. 4. Essentially, the machine is an adaptation of a Scott yarn testing machine. Duplicate jaws were used so that two pieces of tape could be tested simultaneously. By means of reducing gears, the speed of the jaws is brought to 13 in. per min. The two jaws are attached on one threaded shaft driven by a reversible motor so that the jaws can be lowered during the test and raised to the original position after the test at the same rate (13 in. per min.).

The autographic record is drawn on a chart fastened to the drum of a Phipps & Bird Kymograph, the drum of which revolves at the rate of 4 in. per min. The rate of rotation of the Kymograph is not pertinent, although the speed selected was that which would show a reasonable relation to the actual amount of tape stripped from the metal. In practical use, the speed might be greater or less at the desire of the operator. Thus, he could show upon a given chart fewer or more individual tests.

In order to measure the adhesion it was found, after much study, that a push-pull spring balance was the most accurate and most satisfactory means of applying the load. They were made by Chatillon to special design. A lightweight holding device was attached in place of the usual pointer. This carried an arm of spring steel and a pen, light enough in weight so as to be well within the load tolerance of the spring balance. The spring balance was fixed upon a bracket and positioned such that the pull would be exactly vertical. Spring balances of the customary friction-bearing type were wholly unreliable. Therefore, the ones used were made with ball bearings at top and bottom and even these needed to be adjusted with extreme accuracy and the axis of the rod through the balance was made to pass through the exact center of each of the ball bearings. The balances were calibrated frequently by dead weights. Typical calibration charts, taken both by addition and reduction of load, are shown in Fig. 6.

The charts used for these tests were 6 by 20-in. pieces of thin semitransparent paper on which horizontal lines corresponded exactly with the scale markings on the balance. For the 18-oz. balance the chart was ruled in ounces. For the 5-lb. and 10-lb. balances the chart was ruled in quarter and half pounds, respectively, the same chart having been used for either balance. Along the bottom of the charts were spaces for the usual records.

The actual method for performance of the test is as follows:

After the tape is rolled on the metal strip, it remains undisturbed for 15 min. ± 10 sec. Then the tab of the test piece is doubled back over the tape, and the tape stripped by hand up to the first red mark 1 in. from the right end of the metal strip. The end of the metal strip thus uncovered is clamped in the jaw of

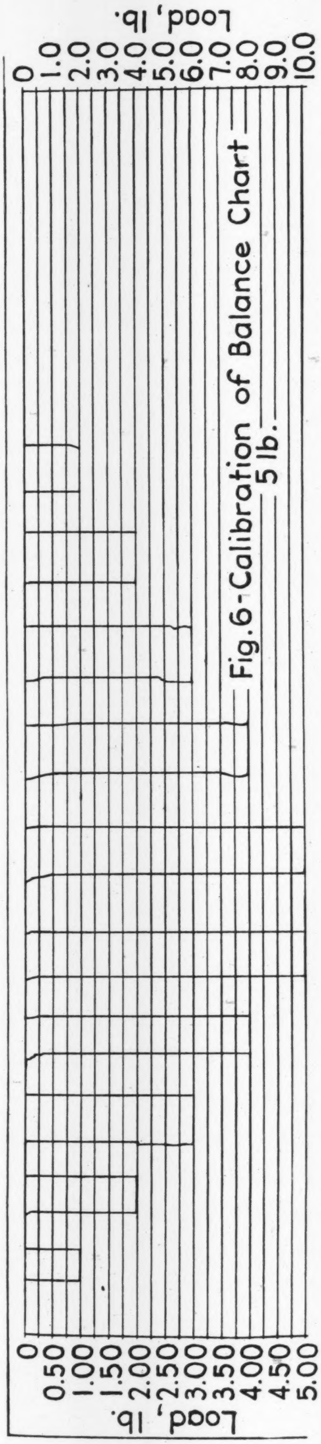

Fig. 6-Calibration of Balance Chart 5 lb.

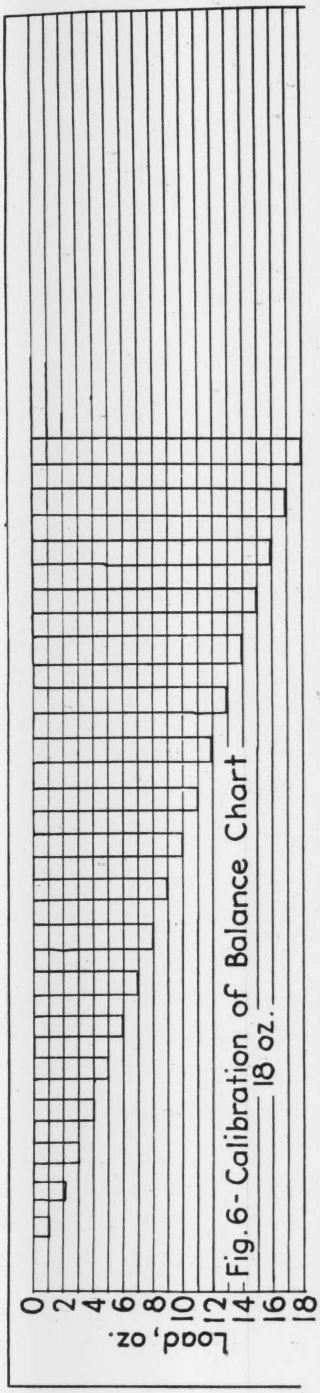

Fig. 6- Calibration of Balance Chart 18 oz.

the testing machine so that the strip is in a vertical position, the tab having been lined up directly in front of the tape. The hole in the upper end of the tab is slipped over a hook at the lower end of the spring balance. Every motion of the operator must be timed in order to duplicate the set-up for each test.

The other test piece is set in position under the second spring balance. When the tape is stripped from the metal piece, the rough paper or cloth backing of the specimen which is still on the metal may rub against the back of the part already being pulled off. The friction thus induced may cause errors in the record. To avoid this, a piece of waxed paper 2 by 8 in. is placed between the tab and the tape. Thus, the rough back may rub against this waxed paper and experience showed that the errors due to friction were eliminated.

The chart is adjusted to zero, and the drum rotated to give a $\frac{1}{2}$-in. zero line. The drum is stopped, and the jaw started downward. The tape is allowed to strip up to the second red mark, 2 in. from the lower end of the lacquered metal strip. At this point the drum is started and an autographic curve of the force required to strip the tape from the lacquered metal is recorded.

When the tape has stripped to the third red mark, 6 in. from the lower end of the metal strip, the drum is stopped. This gives a curve which corresponds to 4 in. of stripping under uniform conditions. The jaw motion is reversed, the balance is allowed to return to zero, and a second $\frac{1}{2}$ in. zero mark is recorded by running the drum for a short time. Thus, a record of accuracy of the spring balance and of the setting of the machine is recorded for each test made. The strips are then removed from the machine.

If reference is now made to any one of the records on Fig. 7, it will be seen that the vertical line indicates the increase of tension until the tape begins to strip from the lacquered metal. From then on, the line is irregular in a horizontal direction and shows the value of the adhesion between tape and lacquer.

In performing this test, rolling one pair of test pieces and stripping another pair which had remained undisturbed for 15 min. ± 10 sec. may be alternated so that the testing may be performed without delay through a given series of tape samples.

For measurements of other qualities of the tape such as the anchorage of the adhesive mass to the paper or cloth backing or the adhesion of the mass to the back a similar technique may be employed.

Stripping from the full roll was performed by the use of a Scott rubber testing machine, the jaw of which was modified by the addition of a metal cylinder 1 in. wide and of such a diameter as to fit inside the cardboard spool of a roll of tape snugly enough to prevent the spool from slipping on the cylinder. This cylinder was mounted in such a way that it could be revolved freely on its axis; see Fig. 5.

The set-up consisted of a Chatillon push-pull spring balance and a Kymograph like those which have been described above. The jaw of the machine was lowered at the rate of $25\frac{1}{2}$ in. per min.

Six feet of tape were cut from the roll and rejected. The roll was placed on the metal cylinder of the test machine so that when the tape was pulled upward from the roll, the back of the tape was toward the operator. A 5-in. length of tape was stripped from the roll. A metal tab $1\frac{1}{4}$ by $2\frac{3}{4}$ in. punched at one end, was fastened to the adhesive $1\frac{1}{2}$ in from the end of the tape, the punched end of the tab being toward the end of

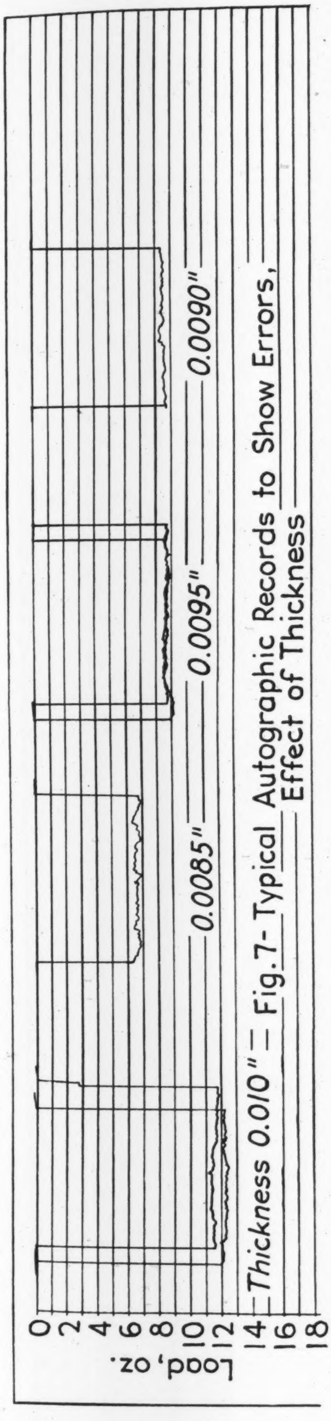

Fig. 7- Typical Autographic Records to Show Errors, Effect of Thickness

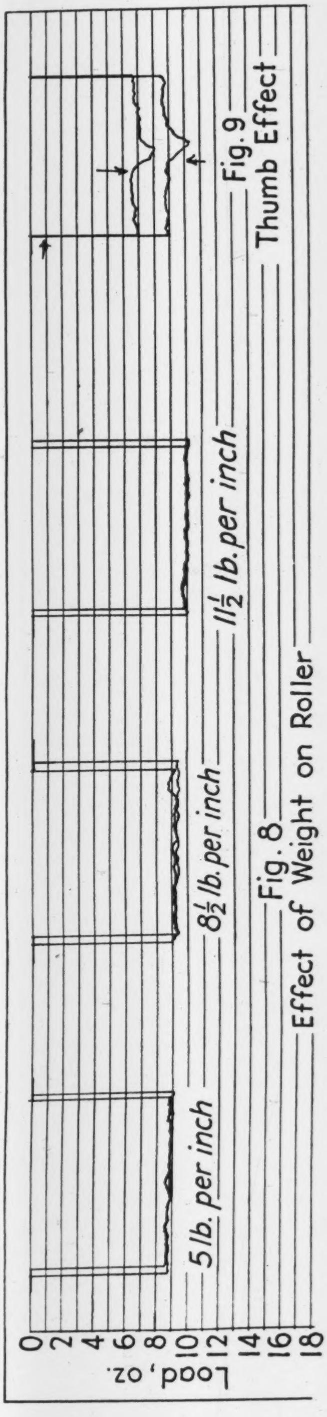

Fig. 8 Effect of Weight on Roller

Fig. 9 Thumb Effect

the tape. The $1\frac{1}{2}$ in. end of the tape was folded over the tab and fastened.

A hole was punched in the tape coinciding with the hole in the tab. On the back of the tape on the tab was written the tape sample number and the roll number. At the juncture between the loose end of the tape and the roll, a red pencil mark was made both on the roll and on the back of the loose end of tape, thus marking off one complete revolution of the tape on the roll. The hole of the tab was slipped over the hook on the bottom of the spring balance.

The chart was adjusted to zero and the drum rotated to give a $\frac{1}{2}$ in. zero line. The drum was stopped and the jaw started downward. As soon as stripping began, the drum was started and an autographic curve of the force required to strip the tape from the roll was recorded.

When the tape had been stripped to the second red mark, that is, one complete revolution of the roll, the drum was stopped. The jaw was raised, the balance was allowed to return to zero, and a second $\frac{1}{2}$ in. zero mark recorded by running the drum from a short time. The test piece was cut from the roll and the tab removed. In performing this test, one roll was prepared and tested at a time.

RESULTS

A very large number of tests were performed upon metal strips lacquered with Duco and other types of automobile finishes. Surfaces such as spring steel, cold-rolled steel, chromium plate, glass, and others were studied. Also the effect of roughening the surface with sand paper and the effect of different methods of cleaning the strips were tested. A discussion of these variables, however, would require an inordinate amount of space. Therefore, only certain results with adhesion to lacquered metal have been reproduced. These have been selected to illustrate not only typical curves, but also to show the effect of some factors which influenced the adhesion to the most pronounced degree.

Figure 7 shows records in which the effect of the thickness of paper-backed masking tape against a Duco lacquer metal strip is recorded. The paper itself was 0.007 in. thick so that the thickness of the adhesive may readily be calculated. The thicker the adhesive mass, the higher the adhesion.

The weight upon the roller was varied and the effect is shown in Fig. 8. In this instance, the heavier the weight, the higher the adhesion. In the case of masking tape which was known to be irregular of face, this merely means that there was a larger area of contact of the adhesive to the lacquer.

Figure 9 is interesting in that these two tape samples were rolled not with the roller, but by the usual method of rubbing them down with the thumb. The irregularities are not only over the entire distance of stripping, but also the higher adhesion when the thumb was allowed to dwell for an appreciable length of time upon part of the tape.

Figure 10 shows the effect of dwell; namely, stopping the roller at a specific point upon each of the samples tested, as is shown, for 10 sec. in one instance and 30 sec. in others. It was results of this sort which influenced the design of the rolling machine, Fig. 1, for it proved the necessity of uniformity of application of load from end to end of the strip.

Figure 10 (*d*) indicates also how the adhesion is reduced by minor impurities upon the surface of the lacquer. In this particular instance, the lacquered strips had been previously used but had not been recleaned.

Figure 18 demonstrate the influence of irregularities of load application.

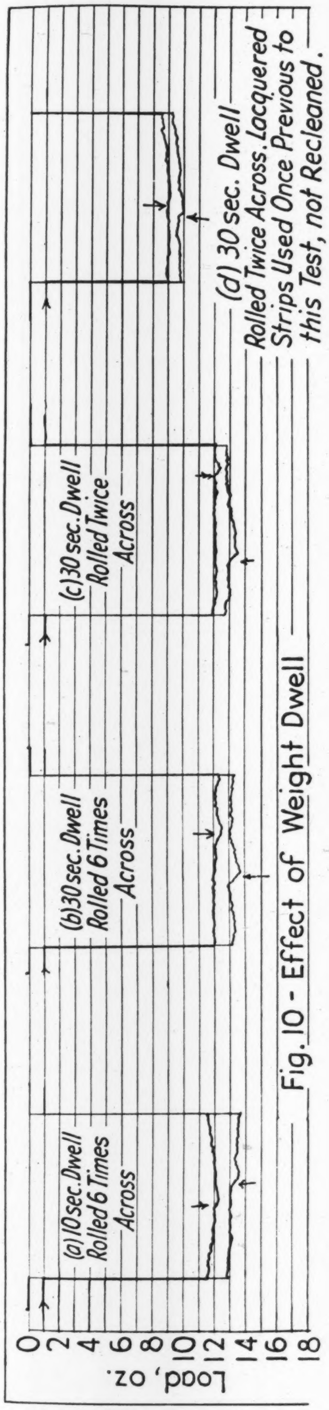

Fig. 10 - Effect of Weight Dwell

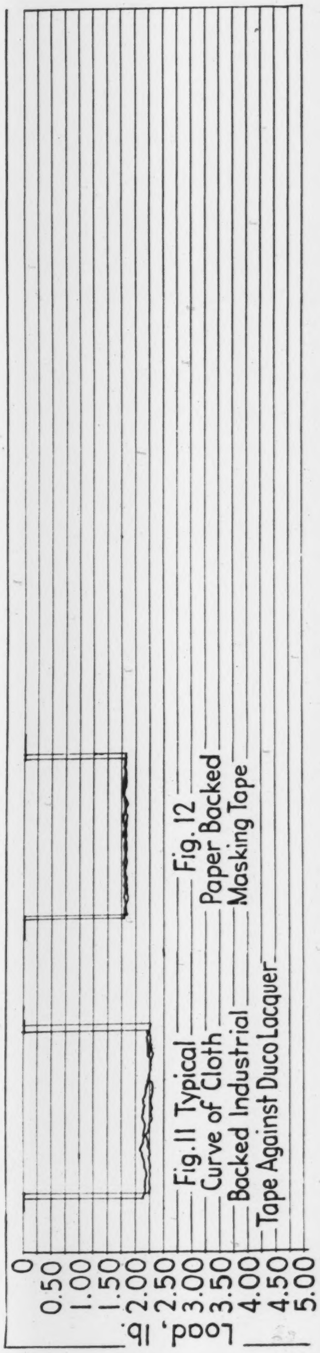

Fig. 11 Typical Curve of Cloth Backed Industrial Tape Against Duco Lacquer

Fig. 12 Paper Backed Masking Tape

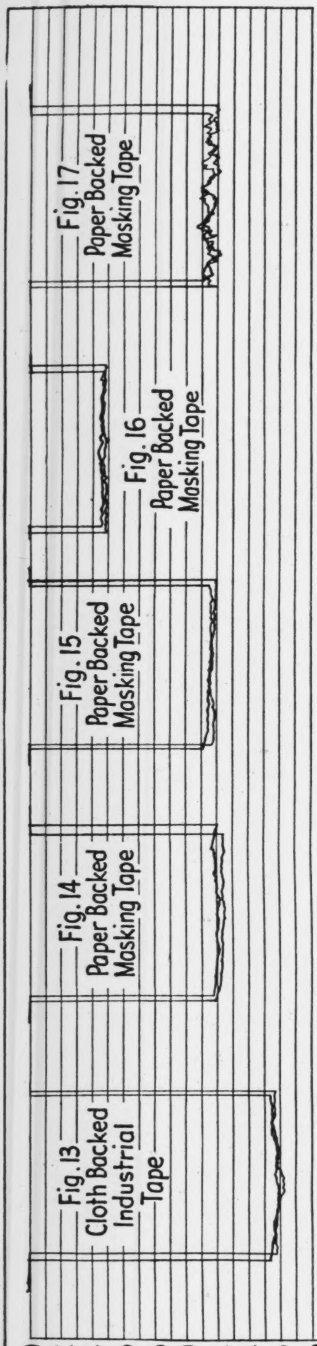
Fig. 13—Cloth Backed Industrial Tape

Fig. 14—Paper Backed Masking Tape

Fig. 15—Paper Backed Masking Tape

Fig. 16—Paper Backed Masking Tape

Fig. 17—Paper Backed Masking Tape

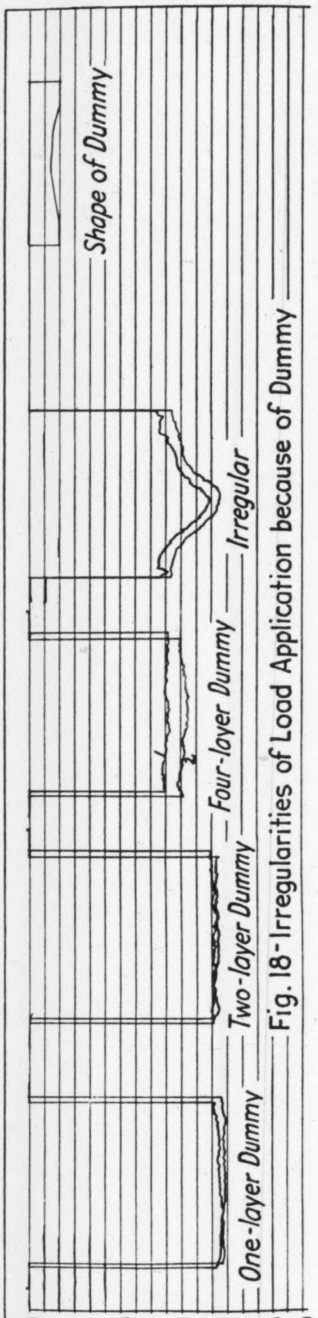

Fig. 18—Irregularities of Load Application because of Dummy

Such tests as these afforded evidence which led to the procedure of always rolling two strips of tape simultaneously, and these two strips of tape being from the same roll and applied upon lacquered metal strips of accurately gaged and identical thickness.

To insure uniformity of pressure of the roller over the tape width two specimens are always rolled at the same time. In Fig. 18, the word "dummy" is used merely to indicate that one or more layers of masking tape were used upon lacquered metal strips in place of a regular strip of tape made up for test. Thus, if the dummy were of one layer only, we get the first curve at the left. When of two layers, the adhesion is somewhat less although the duplicates are somewhat irregular. When in 4 layers, the adhesion and the duplicates are not only less, but differ from each other. When a dummy or any strip of tape is used the thickness of which is greater or less than that of the test specimen, the roller rides at an angle on the test specimen. The pressure, therefore, is not uniform over the width of the tape under test and the curve does not record a true measure of the adhesion. In the case of the fourth curve, a specifically irregular dummy was cut to the shape shown, with the result that the adhesion curve shows somewhat the inverse shape of the dummy.

Because this paper primarily is a description of method and apparatus, only a few copies of curves drawn by tape tested under these conditions are included. The figures mentioned in the following paragraphs are copies of curves selected from a very large number. The discussion of them is necessarily brief.

Drawn on the 5-lb. scale, Figs. 11 and 12 are curves of cloth-backed industrial tape and paper backed masking tape.

Figures 13 to 17 are records of adhesives against Duco lacquered metal strips. The chart and spring balance used were of the 18-oz. scale. The kind of backing is indicated on each figure.

Figure 13 is a standard cloth-backed industrial tape, although it was spread with a different adhesive than that shown in Fig. 11.

Compare Figs. 13, 15, and 17, which show not only the number of ounces of pull required to strip these tapes, but also the uniformity or the irregularity of the curves. When the thickness was the same, we found that each adhesive drew a characteristic curve.

Of particular interest to the authors was a long series of tests of paper-backed adhesive masking tapes after they had been run under specific and uniform conditions of aging. Some rolls were aged in a dry oven and others in a humid oven. By the application of these methods and apparatus, it was possible to appraise the expected life of tapes of different construction and which used adhesives of different compositions.

CONCLUSIONS

The authors performed several thousand tests upon adhesives of different compositions, manufactured under excellent controlled factory conditions and carried upon backs of a variety of constructions. One conclusion was clear, which is included here even though this particular paper is devoted largely to a description of the test procedure alone. This is that other things being the same (paper or cloth of the same construction, methods of manufacture, and so on) each adhesive, when tested as described, gives a curve which is characteristic of its composition.

Acknowledgments:

The authors wish to acknowledge the cooperation of Anthony Hunt, who was an assistant in the laboratory and who performed by far the majority of the

tests. We would like to thank the officials of Johnson & Johnson, of New Brunswick, N. J., for whom the work was done, not only for their uniform cooperation, but for permission to publish this paper.

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MECHANICAL TESTS OF CELLULOSE ACETATE

By William N. Findley[1]

Synopsis

Mechanical properties of cellulose acetate as obtained by tests carried on at constant temperature and relative humidity are presented. The tests consist of short-time extensometer tension tests, long-time constant load (time-to-fracture) tension tests, and vibratory bending fatigue tests of notched and unnotched specimens. A wide range of speeds of testing were used in the short-time tension tests. A critical speed of testing in tension was found above which the stresses were substantially unaffected by changes in speed of testing. The constant-load tension tests show that the fracture stress and the ductility decreased as the time to fracture increased. The effect of the molded surface, the shape of specimen, and notches on the endurance limit in fatigue is shown; and the effect of air velocity and stress upon the temperature of the specimen during repeated stressing in fatigue is indicated. The endurance limit was affected markedly both by the shape of the specimen and by a V-notch.

The investigation reported in this paper was undertaken to secure data on the load-resisting properties of plastics and to study the technique of testing plastics. This technique involves the consideration of variables such as the effect of small changes in temperature and humidity, the effect of speed of testing, the effect of surface condition, and the effect of attachment of instruments. There has been some work done on the mechanical properties of plastics in which certain of these effects have been taken into account. Two papers[2] along lines similar to that discussed herein have come to the attention of the author.

The material selected for this study (cellulose acetate) would not be considered as a structural material for withstanding high stresses; however, a knowledge of its load-resisting properties are needed in the design of smaller but important parts in which the material does have application.

Material:

The material for these tests was supplied by the Plastics Division of the Monsanto Chemical Co. and is a clear, transparent cellulose acetate (Monsanto formulation number 2050 TV). It is composed of medium viscosity cellulose acetate of the acetone soluble type, plasticized with about 26 per cent of phthalate and aromatic phosphate ester plasticizers. All specimens were cut from one sheet $\frac{3}{16}$ in. thick which was made by the sheeter process. The finished sheet contains less than 1 per cent of residual solvent and water, and the molding temperature was between 200 and 250 F.

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[1] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] W. F. Bartoe, "Service Temperature Flow Characteristics of Thermoplastics," *Mechanical Engineering*, Vol. 61, No. 12, December, 1939, p. 892.

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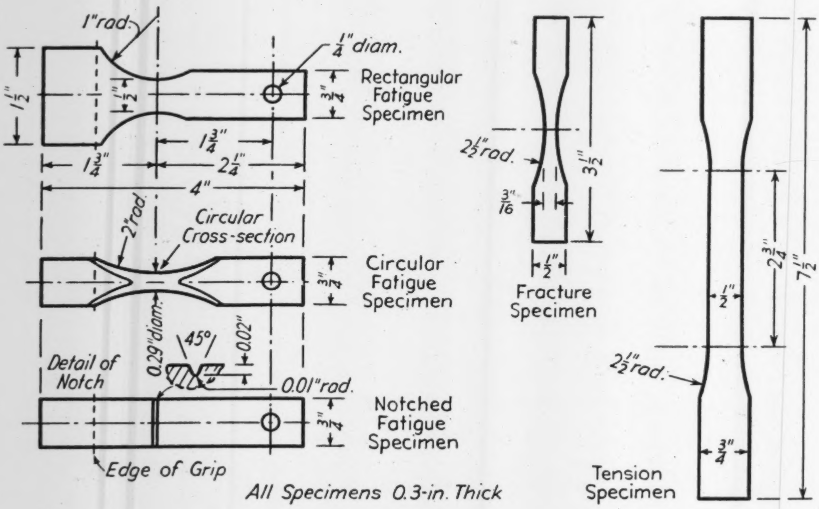
The Rockwell hardness of the material is about L 40 (at 77 F., 50 per cent relative humidity).

Tests and Test Specimens:

The following tests were made: short-time extensometer tension tests, long-time constant load (time-to-fracture) tension tests, and vibratory bending fatigue tests of notched and unnotched specimens. All specimens were cut on a jig saw from one sheet of cellulose acetate 20 by 50 in. described above. The specimens were cut from the sheet with the same orientation so that the tensile stresses set up during the test were crosswise of the sheet. The final shape of the specimens was obtained by milling and the milled edges were finished with No. 0000 emery paper so as to remove all burrs and all scraches transverse to the direction of tensile stresses. Except as otherwise specified the flat surfaces of the specimens were the original (highly polished) surfaces as they came from the sheeter process.

The specimens were conditioned by allowing them to remain at least four days in a constant temperature and humidity room before being tested. This room, in which all tests were conducted and in which all specimens were stored before testing, was maintained at a constant temperature of 77 ± 1 F. and 50 ± 2 per cent relative humidity. All specimens were carefully weighed on an analytical balance before and after testing except for the short-time tension specimens which were weighed only before testing.


1"rad.
1/4" diam.
Rectangular Fatigue Specimen
1 1/2"
1/2"
3/4"
1 3/4"
1 3/4"
2 1/4"
4"
2" rad.
Circular Cross-section
Circular Fatigue Specimen
3/4"
0.29" diam.
45°
0.02"
Detail of Notch
0.01" rad.
Notched Fatigue Specimen
3/4"
Edge of Grip
2 1/2" rad.
3 1/2"
3/16"
1/2"
Fracture Specimen
2 3/4"
7 1/2"
1/2"
2 1/2" rad.
3/4"
Tension Specimen

All Specimens 0.3-in. Thick

FIG. 1.—Test Specimens.

SHORT-TIME EXTENSOMETER TENSION TESTS

Testing Machine:

The tension tests were made on a 1500-lb. Riehle tension testing machine modified to provide pendulum weighing and variable cross-head speed, and equipped with Templin wedge grips. The speed was changed by means of V-belt pulleys. It appears likely that the relationship between rate of strain and head speed, and certain characteristics of the stress-strain diagram, such as the upper yield point, may be dependent to a large extent upon the rigidity of the machine. Therefore, the spring constant (the ratio of load to deformation of machine and grips) of this machine was determined and found to be 10,170 lb. per in.—a rather "soft" (nonrigid) machine.

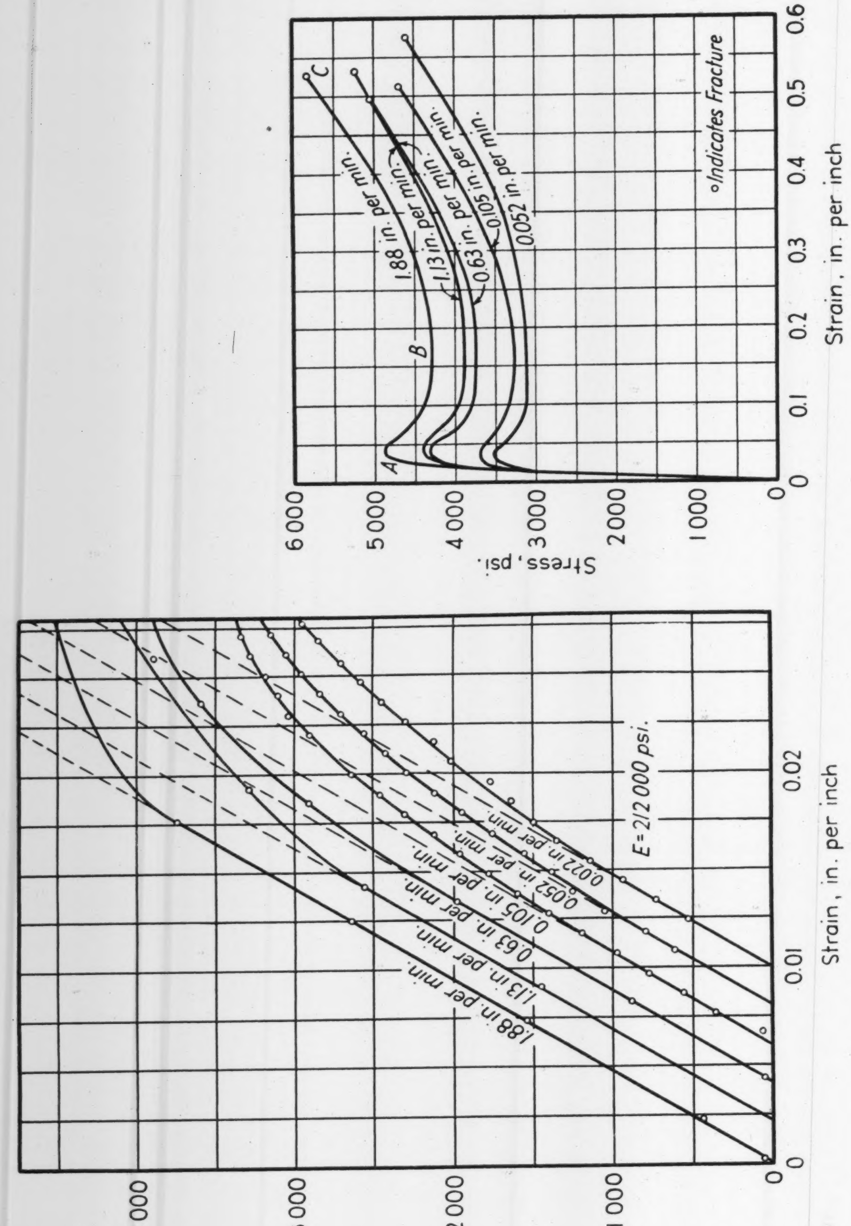
Extensometer:

Since it was desired to obtain the complete stress-strain curve to fracture, it was necessary to construct an extensometer having a very long range, having sufficient sensitivity to obtain satisfactory readings in the elastic range, capable of gripping the specimen even with large reductions in cross-section, and of withstanding the shock of fracture, and having contacts between the instrument and specimen that would not cause fracture to take place at those points. The instrument, evolved after several trials and modifications, was essentially a parallelogram consisting of two long arms pin-connected to a 2-in. bar at one end with the specimen at the other end. A 1-in. travel dial was placed halfway between the ends of the long arms. The dial was pivoted to one of the arms and the plunger of the dial was fastened by a friction grip to an adjusting screw which was pivoted to the other arm of the instrument. The friction grip was used so that the dial would not be damaged if the instrument should be extended beyond its normal limits of travel due to the shock of fracture. During a test the free end of the instrument was suspended from the testing machine and the weight of the specimen-end of the instrument was balanced by means of pulleys and counterweights so that relatively small contact pressure between the instrument and the specimen was necessary to keep the two together. It was found that contact points having a radius of about $\frac{1}{32}$ in. caused enough indentation of the specimen to hold the instrument in place and yet did not cause failure at these marks. The difficulty encountered by the reduction in cross-section of the specimen was overcome by a spring arrangement with sufficient range of motion to keep the contact points pressed against the specimen automatically even for large reduction in cross-section.

Procedure:

Specimens of the shape shown in Fig. 1 were tested in tension for a variety of different no-load head speeds ranging from 0.022 to 4.5 in. per min. During each of the tests simultaneous readings of load and extension were taken; and for some of the tests simultaneous readings of time were also taken. Readings were taken from the start of the test until fracture and an attempt was made to space the readings in such a way as to permit the plotting of a smooth stress-strain curve. However, at the higher speeds it was difficult to obtain readings in the "elastic" region. Autographic equipment is essential to an accurate record of the "elastic" region for head speeds above 2 in. per min.

From the data obtained in this way, the stresses and strains were computed (the stress was in all cases computed from the original cross-sectional area). Then three sets of curves were plotted: stress *versus* strain to a scale large enough to show the "elastic" region (Fig. 2 (*a*)); stress *versus* strain for the entire curve (Fig. 2 (*b*)); and strain *versus* time (Fig. 4). A marked change in the character of the "elastic" region is noticed with increasing head speed. At the lowest speeds—0.022 and 0.052 in. per min.—the stress-strain relation is a curve almost from the start. At higher speeds this curvature disappears and the relationship becomes nearly a straight line. It will be noticed that the initial slope in all cases is the same (the dash lines are all parallel). It seems probable



(a) Elastic region.

(b) Entire curve

FIG. 2.—The Effect of Speed of Testing on the Stress-Strain Relation in Tension.

that the curvature noticed in the tests at low speeds is due to creep which, at the low speeds, takes place at such a rate as to increase materially the strain readings. The fact that the change in the stress-strain relation is from a straight line to a curve rather than simply a change of slope of the straight line is a verification of the well-known fact that the creep rate increases with the stress.

The initial modulus of elasticity was found to be the same for all speeds tested, as may be seen from the dash lines (all of which are parallel) in Fig. 2 (*a*). The value of the modulus of elasticity obtained from the slope of the dash lines is 212,000 psi. The effect of creep at low speeds makes it difficult to obtain enough points at the initial slope to determine the modulus at head speeds below about 0.5 in. per min. (rate of strain in "elastic" region about 0.05 per min., see below).

The curves shown in Fig. 2 (*b*) indicate that the material has a definite "yield point" (point *A* on the curve) and that the general shape of the stress-strain relation is the same for all speeds except perhaps for very slow speeds where creep plays an important part. It is, however, noticeable that the stresses are increased with increasing speed. The exact nature of the effect of head speed upon the tensile properties is more clearly illustrated by Fig. 3 in which are plotted the upper yield point, the lower yield point, the fracture stress, the reduction of area, and the elongation in 2 in. (measured at the instant of fracture) as functions of the no-load head speed of the testing machine. The no-load head speed is used here for convenience. The relation between head speed and the more significant quantity, rate of strain, will be discussed later. The upper yield point is here defined as

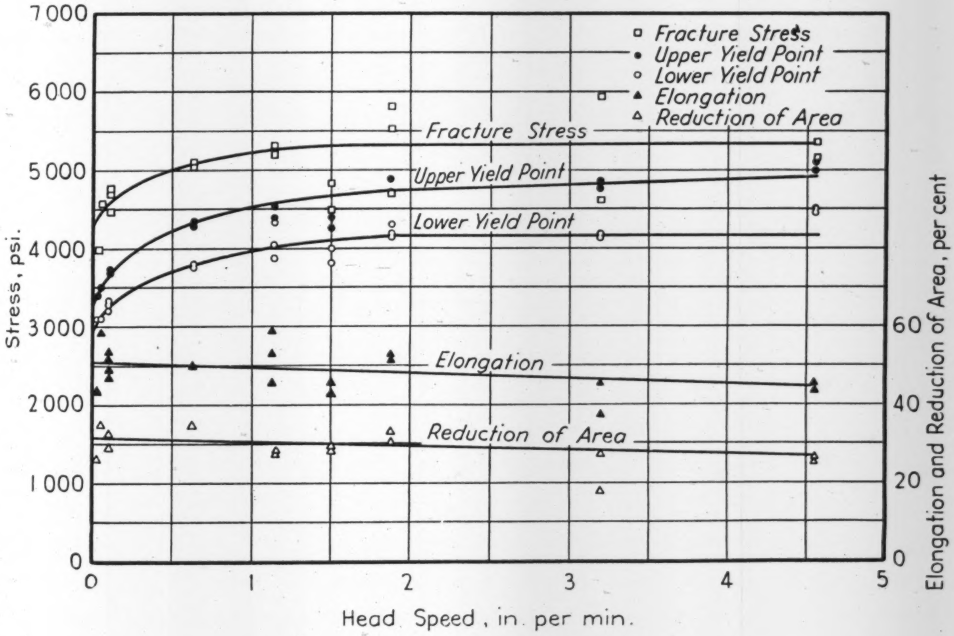


FIG. 3.—The Effect of Speed of Testing on the Tensile Properties.

the stress corresponding to the peak of the curve at the point *A*; the lower yield point is the stress corresponding to the horizontal portion at point *B* at which the strain continues without further increase in stress; the fracture stress is the stress at fracture, point *C*, as computed from the original cross-sectional area; the reduction of area is the final area of cross-section expressed as a percentage of the original area; and the elongation is the change in length at time of fracture of an original 2-in. gage length expressed as a percentage of the original gage length.

Figure 3 shows that the upper yield point, lower yield point, and fracture stress all increase with increasing head speed until a critical speed is reached at about 1.5 to 2 in. per min. beyond which they remain substantially constant. An examination of these data will indicate that the values of the lower yield point are more consistent, as shown by less scatter of the data, than either the upper yield point or the fracture stress. This fact becomes more significant in the light of the following remarks: It was noticed during the course of the experiments that very slight transverse surface scratches formed the nucleus from which the final fracture started and that the presence of such imperfections markedly affected the value of the elongation and the stress at which fracture took place. For this reason great care was taken to provide specimens free from scratches. If the specimens had not been very carefully prepared there would have been more scatter in the data for the fracture stress. Further, it has been shown for mild steel that the magnitude of the upper yield point depends upon the rigidity of the testing machine used. While there are as yet no data to prove that this is also true for cellulose acetate, it seems likely. In the light of these considerations, the lower yield point would seem to be the more significant tensile property of this material since the lower yield point appears to be the least

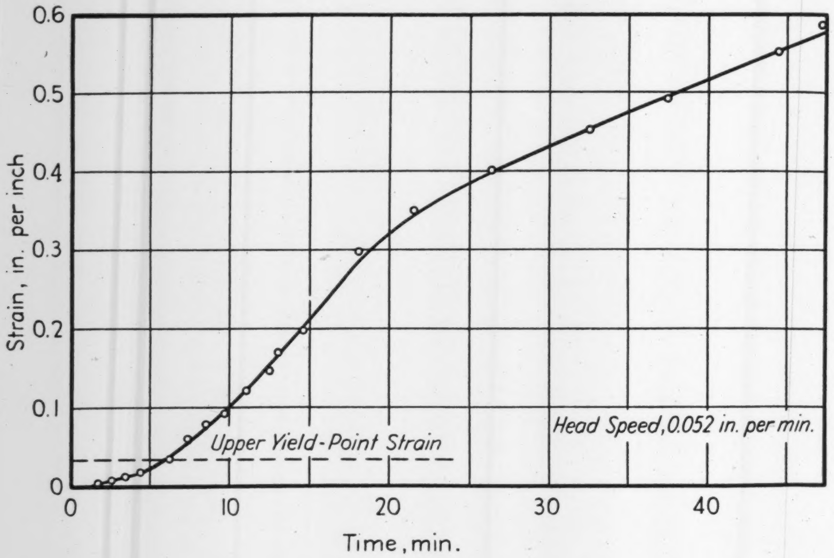


FIG. 4.—Relationship of Strain to Time During a Tension Test.

affected (of the three) by variations in testing conditions. Incidentally, the lower yield point is readily determined at any testing machine speed with any machine in which the load is self-indicating.

Figure 3 also shows the reduction of area and the elongation in 2 in. at fracture plotted against the no-load head speed. It will be noticed that there is considerable scatter of the data. This scatter is perhaps in part due to the fact that the sheet from which the specimens were cut was not of uniform thickness; and since it was desired to leave the original surface on the flat sides of the specimen, this unevenness was not corrected and might account for some scatter in reduction of area and elongation. However, a large part of the scatter may be attributed to slight surface scratches causing early failure. It will be noticed that there is an indication that both reduction of area and elongation decrease with increasing head speed.

In Fig. 4 is shown a sample curve of strain plotted against time. Such data were obtained from specimens tested at several different head speeds. It will be noticed that there is a distinct change in slope of this curve as the region near the upper yield point is passed. This is due to the fact that up to the upper yield point the load is increasing not only on the specimen but also on the testing machine and hence part of the turning of the screw of the testing machine results in deformation of the testing machine. In a relatively non-rigid machine this may be a large part of the total motion of the screws; hence, the rate of stretching of the specimen is less than that which would occur if the machine were more rigid. But when the lower yield point has been reached the motion of the screw results almost entirely in stretching the specimen because there is no longer any increase in the load on the testing machine and hence the machine does not deform further. Thus in the lower yield point region the rate of straining is much greater than the rate of straining up to the upper yield point. It is evident from this discussion that the variation of rate of strain with time depends to a large extent upon the shape of the stress-strain curve of the material being tested when a machine equipped for constant rate of cross-head motion is used.

The relationship between rate of strain and no-load head speed for the "elastic" region was obtained from strain *versus* time curves (similar to the representative curve in Fig. 4) at several different no-load head speeds. This relationship was obtained by plotting the slope of the portion of each curve lying below the upper yield point region against the no-load head speed. A similar study was made of the rate of strain in the region of constant stress. The relationships are: rate of strain in elastic region (per min.) = 0.095 × no-load head speed (in. per min.); rate of strain at constant stress (per min.) = 0.475 × no-load head speed (in. per min.). Hence, by a mere change of scale the abscissa in Fig. 3 would become rate of strain instead of head speed without any alteration in the position of the plotted points.

FRACTURE UNDER LONG-CONTINUED CONSTANT LOAD

The fracture test is used in the study of the behavior of materials that are subject to creep under long-continued steady load. The specimens used in the fracture tests are illustrated in Fig. 1. They were shaped by milling and the milled edges were smoothed with emery paper, but not polished. The apparatus for the tests consisted of a steel rack from which the specimens were suspended and loaded in tension by hanging sufficient weight from the specimen to produce the desired stress. This weight remained hanging on the specimen until the specimen broke at which time the falling of the weight would release a catch to stop a clock and thus record the time of fracture.

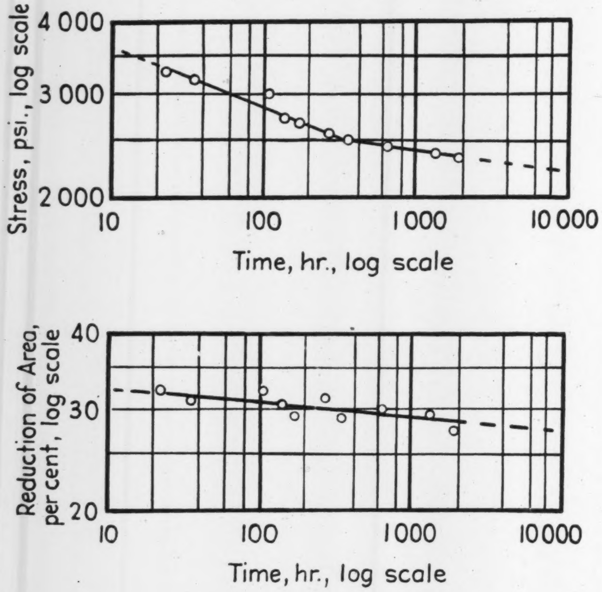


Fig. 5.—The Effect of Time to Fracture Under Constant Load on the Fracture Stress and Reduction of Area.

Effect of Time on the Fracture Stress and the Ductility:

A plot of stress against the time to fracture of cellulose acetate on a log-log scale appears in Fig. 5, and also a plot of the reduction of area against the time to fracture. It will be noticed that there is a sudden change in slope of the stress *versus* time-to-fracture curve at about 300 hr. The fracture stress at 1000 hr. (about one month) is 2430 psi. Extrapolating the data as a straight line with slope corresponding to the lower portion of the curve the fracture stress at 10,000 hr. (about one year) is 2200 psi., which is about half the fracture stress obtained in short-time tension tests. An indication that the ductility, as measured by the reduction of area, decreases with increasing time to fracture is shown in Fig. 5. From these data it would seem that consideration should be given to the decrease in fracture stress with increase in time to fracture when cellulose acetate is to be subjected to long-continued steady load.

FATIGUE

Five sets of fatigue tests of cellulose acetate have been run to determine the effect of the molded surface, the effect of shape of specimen, and the notch sensitivity. A Krouse plate fatigue machine, which is a vibratory type of machine designed to produce the same maximum deflection of the specimen in each cycle, was used. In these tests, the machine was not equipped with variable speed and hence the tests reported were made at a constant rate of 1720 cycles of stress per min. During the first few tests of this series the specimens frequently broke in the grip at either end even though some allowance had been made in the design of the specimen for the stress concentration at the grips. The cause of the difficulty seems to have been a combination of stress concentration and the chafing of the specimen resulting from the minute sliding motion between specimen and grip which occurs at the edge of the grip during the repeated bending. This difficulty was cured by placing a $\frac{1}{16}$-in. sheet of rubber gasket between specimen and grips. This type of machine requires that the specimen be calibrated as a cantilever beam to determine the load-deflection relationship from which

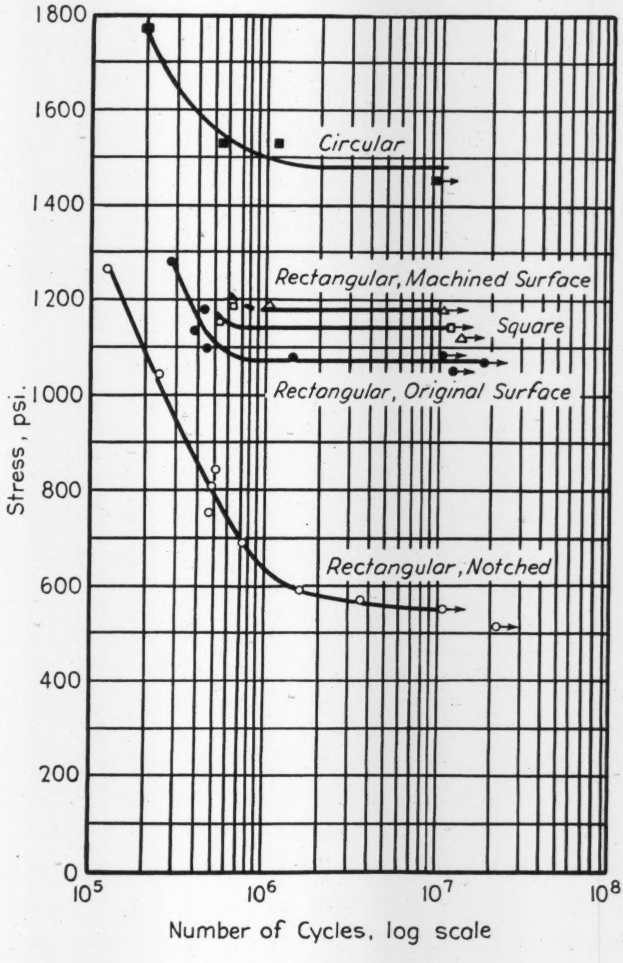


FIG. 6.—*S-N* Diagrams.

the deflection necessary to produce the desired stress can be obtained. It is quite noticeable during the calibration that creep occurs as evidenced by increasing deflection under constant load. However, it was found that very satisfactory straight line load deflection curves could be obtained if the calibration was made quickly and the load removed after each reading of the deflection.

Figure 6 shows the results of the five series of tests. This diagram is a semi-log plot of stress *versus* number of completely reversed cycles of stress required to cause fracture (the so-called *S-N* diagram).

The specimen used in the first series of tests is shown in Fig. 1. It was of rectangular cross-section (width $\frac{1}{2}$ in., depth $\frac{3}{16}$ in.) in which the original polished surface remained on the flat faces of the specimen and the neutral surface of bending of the specimen was parallel to the plane of the figure. This latter was true of all other fatigue specimens shown in Fig. 1. It will be noticed that the *S-N* diagram for this test, and indeed for all other diagrams shown, has a definite sharp break at about one million cycles. This indicates that a "true" endurance limit has been found, that is, a value of completely reversed stress above which fracture of the specimen will occur for a finite number of cycles of stress but below which fracture will not occur for an indefinitely large number of cycles of stress. The value of the endurance limit for this shape specimen was found to be 1075 psi.

Effect of Molded Surface:

A second set of specimens was tested having the same general proportions as the first but with the original surface machined off to a depth of 0.025 in. Tests of these specimens under the same conditions as the first group gave an endurance limit of 1180 psi., an increase of 9.8 per cent over the endurance limit of the specimen with the original surface.

Effect of Shape of Specimen:

The effect of shape of specimen was investigated in the next two series of tests. In one of these the width of the test section of the specimen was reduced so as to give a square cross-section. In this specimen the original surface remained on the specimen. The endurance limit found was 1145 psi., an increase of 6.5 per cent over the endurance limit of the rectangular specimen with the original surface. In the other series of tests a specimen of circular cross-section as shown in Fig. 1 was used. The endurance limit obtained for this specimen was 1480 psi., an increase of 25.4 per cent over the endurance limit of the rectangular specimen with machined surface. The comparison is made with the machined surface specimen because the test section of the circular cross-section specimen was also machined. With cellulose acetate, as has been found for other materials, the shape of specimen seems to have a distinct effect upon the endurance limit under repeated stresses.

Notch Sensitivity:

The notch sensitivity in flexural fatigue of the material being tested was determined by obtaining an endurance limit of specimens across one face of which a V-notch had been machined as shown in Fig. 1. The endurance limit as computed from the elementary flexure formula using the reduced cross-section at the notch was 550 psi., a reduction of 53.4 per cent from the endurance limit of the rectangular machined specimen. The effective stress concentration factor for a 60-deg.

V-notch (with 0.01-in. radius) in the material being tested was thus found to be 2.15, as obtained from the ratio of the endurance limits of the notched and notch-free specimens. The notched specimen was compared with the rectangular machined specimen because the material which is tested in the notched specimens is that at the root of the notch and hence is equivalent to the material tested in the machined specimen.

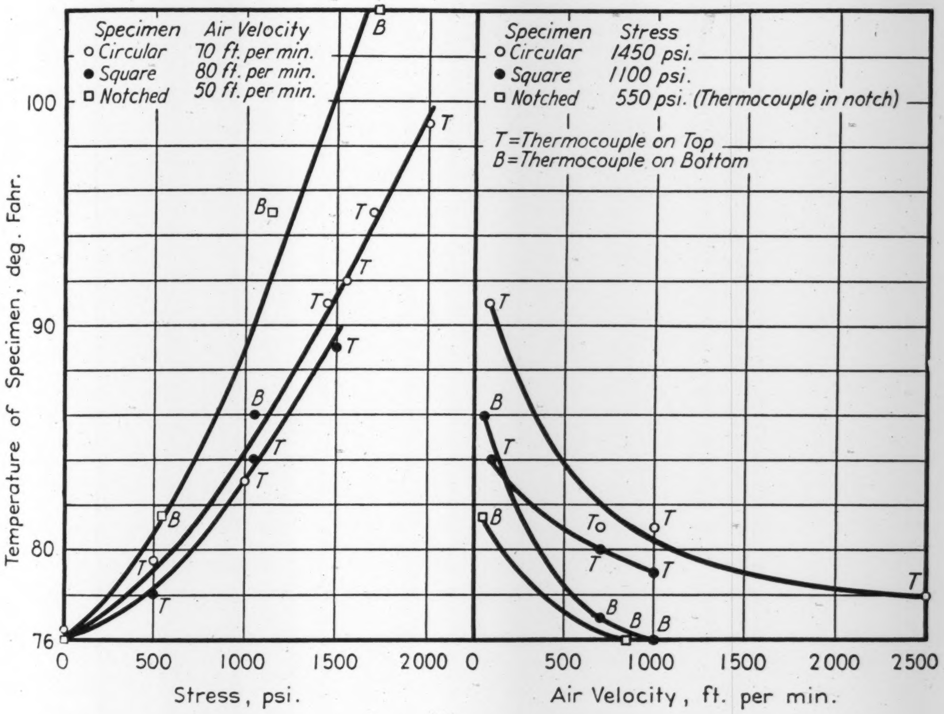


FIG. 7.—The Effect of Stress and Air Velocity on Specimen Temperature in Fatigue.

Effect of Air Velocity and Stress upon Specimen Temperature:

At the conclusion of three of the fatigue tests in which specimens "ran out," the temperature of the high stress portion of the specimen was measured during the operation of the machine by affixing a chromel-alumel thermocouple to that point on the specimen by means of a small strip of cellulose tape. The temperature in each case was found to be above room temperature, which is not surprising when it is recalled that cellulose acetate has rather large damping capacity and hence would absorb considerable energy under repeated stressing. Also the thermal conductivity of the material is low so that the heat could not be readily dissipated. The variation of test-section temperature with stress and also with the velocity of air movement over the specimen is shown in Fig. 7. It will be noticed that the temperature of the notched specimen at the root of the notch is higher than the temperature of the other shapes for corresponding stresses. This is to be expected since it is known that the effect of the notch is to produce higher stresses than that predicted from the elementary theory of flexure. At the endurance limit of both notched and square specimens an air velocity of 1000 ft. per min. was sufficient to reduce the specimen temperature to room temperature, at least on the side receiving direct impingement of the air blast from the fan. All specimens were weighed on an analytical balance both before and after fracture. In all cases the weight was found to have decreased 0.1 to 0.3 per cent. This may be due to the effect of the increased temperature. However, no detailed study of these data has been made. It is proposed to obtain endurance limits for specimens whose temperature is maintained at room temperature, but time has not permitted the completion of this work.

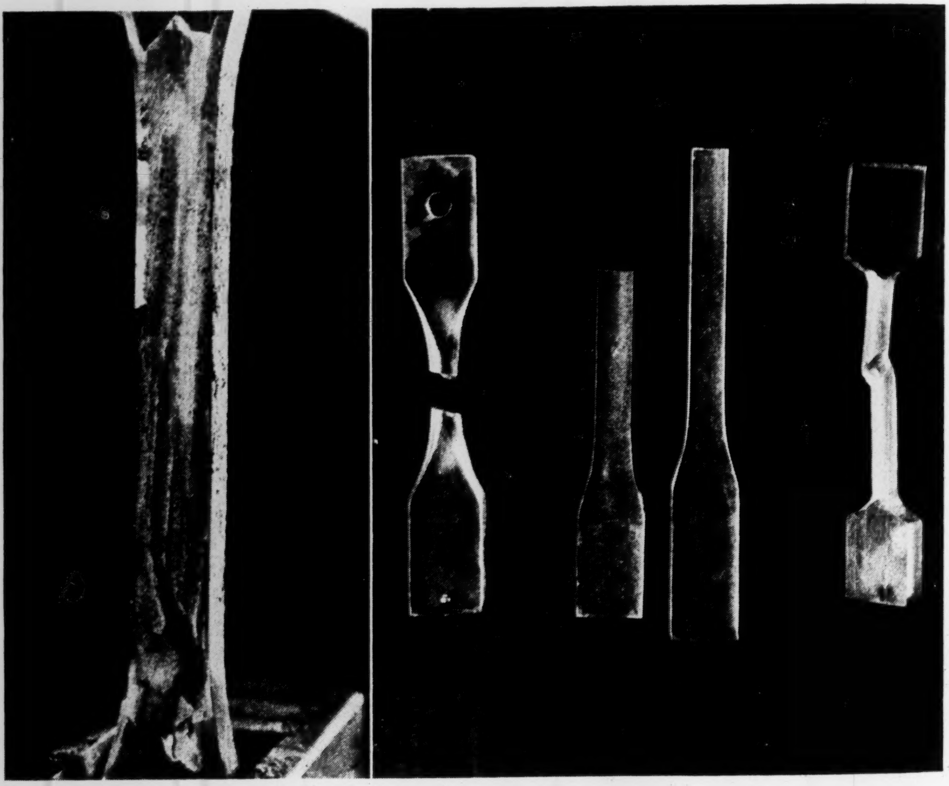

(a) (b) (c) (d)

FIG. 8.—Specimens and Fractures.

(a) Lime-coated tension specimen.
(b) Fractured circular cross-section fatigue specimen.
(c) Fractured tension specimen.
(d) Fractured torsion specimen.

MODE OF FAILURE OF CELLULOSE ACETATE

Figure 8 shows the unusual (as far as metals are concerned) character of the fracture for several fractured specimens. During the progress of the tension test a considerable elongation and reduction in cross-section of the specimen takes place. The character of the tension failure is unlike that occurring in any single metal in that both brittle and ductile properties are evidenced. The large elongation is evidence of ductility yet it is not (as it is in metals) the result of sliding along planes of maximum shearing stress (at 45 deg.) as may be seen from the lime-coated specimen, Fig. 8(*a*). It will be noticed that the lime cracked in transverse lines, beginning first at the corners of the specimen. Ductile steel would have shown diagonal cracks (Lüders lines) in such a coating. Further, there is no marked localized reduction of area (necking down); the reduction is uniform throughout the test section. The fracture is a transverse plane which is characteristic of the tension fracture of brittle materials, Fig. 8(*c*). The fracture starts, perhaps at several places at once, as a tear originating at some slight surface imperfection which spreads leaving a shiny surface until rupture takes place. The surface of rupture shows a fibrous character.

In the fatigue tests, on the other hand, the fracture is propagated in a different manner. In the case of the circular specimens the plane of fracture was at 45 deg. to the plane of maximum normal stress indicating a shear failure as shown in Fig. 8(*b*). Fracture of the rectangular specimens started in the same way but due perhaps to the shape of the specimen the fracture was a slowly propagating tear having the appearance of a glass-like fracture.

One single specimen has been tested in torsion and is shown in Fig. 8(*d*). While there were no data taken on torque or twist, the test is of interest because of the fact that the specimen shown twisted around $1\frac{3}{4}$ revolutions before fracture started. This indicates large shearing deformation, but the fracture was on a 45-deg. helix (a plane of maximum tension stress). Thus again, ductility is indicated by the large detrusion whereas the fracture is that characteristic of brittle material.

Acknowledgments:

Acknowledgment is made to F. B. Seely and H. F. Moore for their help and suggestions during the course of these tests and the preparation of this paper. A portion of the work reported was performed as a senior thesis in the College of Engineering of the University of Illinois by Kenneth Young under the direction of the author. The tests reported are a part of the work of the Engineering Experiment Station of the University of Illinois, M. L. Enger, Director, in the Department of Theoretical and Applied Mechanics of which F. B. Seely is head. Acknowledgment is also made to the Plastics Division of Monsanto Chemical Co. for the material supplied, and to the U. S. Soybean Laboratory at the University of Illinois for the loan of certain equipment.

DISCUSSION

MR. G. M. KLINE.[1]—I want to ask Mr. Findley about the curve which he showed for long-time tension on this cellulose acetate plastic (Fig. 5). He calls particular attention in the paper to the break in the curve at 300 hr., and I wonder whether those points had been established by just one test or whether they were the average of several tests at the particular times involved. In other words, I do not believe there is any reason to expect the sharp break, and it is possible that it may be fortuitous rather than an actual break in the curve.

Also, I want to stress the point that he brought out on the increase in temperature of the plastics when they are subjected to rapid alteration of stress. That damping property of plastics has been stressed in calling attention to some of their advantageous properties and it is a desirable feature from the standpoint of resistance to rupture under conditions involving vibration. But on the other hand, when the vibrations become excessive, as they may in certain airplane applications, that property becomes a disadvantage. As Mr. Findley points out, the energy absorbed has to go some place, and it comes up in the form of heat. That has two serious features. In the first place, there may be sufficient heat generated actually to deteriorate the plastic or to char it. In the second place, as has been pointed out in other papers presented before the Society, the modulus characteristics of plastics decrease with increasing temperature, and one begins to get an accelerated effect on the flow of the material. Even on thermosetting plastic materials, a stretching or flow of the plastic is obtained, which can be very disadvantageous.

So it is well to keep that property in mind in working with plastics for applications where a very high frequency of vibration is involved, and where there may be a very considerable building up of heat, particularly internally. Plastics are also poor conductors of heat, and simply the fact that there is an air current blowing over the surface may not be sufficient to conduct the heat away from the interior of the article.

THE CHAIRMAN (*Mr. L. M. Currie*[2]).—I do not know whether others have noted it in reading this paper, but I think it highly significant that it was found that by removing the surface—the original surface of some of these samples—the endurance limit in fatigue was stepped up almost ten per cent. I wonder how that checks with the experience of others who have been running fatigue tests. What is the effect of the removal of the skin? Mr. Findley says that that figure was checked, but to me it was a unique fact that the removal of the skin of the original finish actually increased the fatigue value.

MR. WILLIAM N. FINDLEY[3] (*author's closure*).—Mr. Kline raises some questions regarding the long-time steady load (fracture) tests, Fig. 5, and interpretation of the results.

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[2] Superintendent, Vinylite Division, Bakelite Corp., Bound Brook, N. J.

[3] Instructor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

The plotted points do represent single tests, but it is the opinion of the author that the loads, areas and times involved in these tests were large enough to permit accurate measurement, and that the uniformity of the material tested as shown in all of the tests was quite satisfactory. The fact that a change in slope of the stress *versus* time-to-fracture curve was observed does not, of course, prove that an "endurance limit" exists for long-time steady loading of cellulose acetate such as is found for repeated loading, Fig. 6. Such proof demands further study. It is possible that the change in slope of the "fracture" curve (Fig. 5) may be due in part to aging (a changing in mechanical properties with time as a result of changes in moisture and solvent content, or progressive polymerization) since the test extended over a period of three months.

It was not the intention of the author to imply either in the text or in Fig. 5 that the change in curvature was instantaneous but to call attention to the fact that a change in slope occurred.

Mr. Kline's statement that the high damping capacity of plastics "is a desirable feature from the standpoint of resistance to rupture—under vibration," may give one the impression that high damping capacity improves the resistance of the material to rupture under vibration. The actual effect is of course the reverse, as Mr. Kline points out later. A large damping capacity does, however, enable the material to absorb energy, thus preventing the amplitude of vibration at a resonant frequency from building up. As a result, the stresses developed are smaller.

I believe that the effect of heat resulting from repeated loading of plastics is not quite as serious as Mr. Kline has pictured it. A plastic may deteriorate as a result of heat developed by repeated loading in the sense that its resistance to fatigue may be lowered by a small rise in temperature. However, it is unlikely that a plastic part would ever char as a result of heat generated by repeated loading at stresses below the room-temperature endurance limit of the material.

Types of loading such as flexure or torsion produce stresses which are maximum at the surface of the part so that the greatest amount of heat resulting from repeated loading of this type is developed at the surface where it may be removed by convection. This fact helps to offset the disadvantage (as far as removal of heat is concerned) of the low thermal conductivity of plastics.

Mr. Currie asks whether the effect of the removal of the skin of the material on the endurance limit was confirmed. I can only say that the increase in endurance limit with removal of the surface was observed but that aging may have been a factor since the fatigue tests reported extended over a period of six months.

RESISTANCE OF PLASTICS TO CHEMICAL REAGENTS

By G. M. Kline,[1] R. C. Rinker,[1] and H. F. Meindl[2]

Synopsis

The results of tests of the resistance of various types of plastics to chemical reagents made in accordance with A.S.T.M. Tentative Method of Test for Resistance of Plastics to Chemical Reagents (D 543 - 39 T)[3] are reported. The plastics examined were as follows: molded, cast and paper-base laminated phenol-formaldehyde resin, molded and paper-base laminated urea-formaldehyde resin, molded and cast polystyrene, cast methyl methacrylate resin, vinyl chloride-acetate resin, vinyl butyral resin, cold-molded bituminous plastic, cold-molded phenolic plastic, cellulose nitrate, cellulose acetate, ethylcellulose, and casein plastic. The resistance of these materials to all of the standard and supplementary reagents listed in the A.S.T.M. method was determined. These reagents include weak and strong acids, weak and strong alkalies, salt solutions, hydrogen peroxide, and organic solvents. Changes in the weight, dimensions, and appearance of the test specimens are recorded.

In the tabulation of data on plastics for handbooks and manufacturers' bulletins, information regarding the effect of various chemical reagents on these materials is generally included. The selection of compounds and concentrations for use in such tests was given early consideration by the Society's Committee D-20 on Plastics, and the Tentative Method of Test for Resistance of Plastics to Chemical Reagents (D 543 - 39 T)[3] was included in the first group of methods developed by that committee. This provided not only a uniform test procedure for use in purchase specifications, but also a guide to investigators so that data obtained on new plastics would be comparable with those in the literature for other plastics.

The limitations of this method as well as of other test methods involving the permanence properties of plastics should be recognized. The choice of reagents typical of the range of acids, alkalies, salt solutions, solvents and other chemicals which may be encountered in service is necessarily arbitrary. For most applications it will be necessary to test the plastic, which is under consideration for fabricating the part, in contact with a particular chemical. However, rather than confine the method to a statement of testing procedure without listing any reagents or extend the list indefinitely to include all compounds and concentrations of solutions proposed by committee members, an effort was made to choose those reagents which would be representative of strong and weak acids, strong and weak alkalies, salt solutions, and the various types of organic solvents, such as alcohols, ketones, esters, and aliphatic, aromatic, and halogenated hydrocarbons.

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[2] Chemical Engineer, Ammonia Dept., E. I. du Pont de Nemours and Co., Inc., Belle, W. Va.

[3] 1939 Book of A.S.T.M. Standards, Part III, p. 805.

Another limiting factor in the practical value of the results obtained in this test is the relatively short time of immersion specified: namely, 7 days. For applications involving continuous immersion in chemicals, the data obtained in a short-time test are of interest only in eliminating the most unsuitable materials. Furthermore, the criteria of changes in weight, dimensions, and appearance are often insufficient to reveal adverse effects of the chemicals on the strength and electrical properties of the plastics. On the other hand, many instances have been reported of industrial applications of plastics in which the surface appearance is marred by contact with chemicals, but the serviceability of the plastic is not appreciably reduced. The period of immersion and the properties to be measured were, as in the case of the chemicals, selected primarily to serve as a guide to investigators wishing to compare the relative resistance to chemicals of different plastics. Those with special problems in the application of plastics under corrosive conditions must necessarily fit to their special needs the duration of contact with the chemical, the temperature of the system, and the physical tests used in determining the suitability of the plastic.

TABLE I.—THICKNESSES AND SPECIFIC GRAVITIES OF THE PLASTICS EMPLOYED IN TESTS.

Material	Thickness, mils	Specific gravity
Phenol-formaldehyde molded	128	1.40
Phenol-formaldehyde cast	127	1.29
Phenol-formaldehyde laminated	134	1.33
Urea-formaldehyde molded	125	1.47
Urea-formaldehyde laminated	132	1.46
Vinyl chloride-acetate resin	133	1.35
Vinyl butyral resin	122	1.09
Methyl methacrylate resin	139	1.16
Styrene resin (molded)	126	1.03
Styrene resin (cast)	156	1.03
Cellulose nitrate	123	1.36
Cellulose acetate	133	1.28
Ethylcellulose No. 1	185	1.10
Ethylcellulose No. 2	166	1.12
Cold-molded phenolic	128	2.09
Cold-molded bituminous	130	1.87
Casein	121	1.25

The experimental data reported in this paper were obtained in an exploratory investigation undertaken by Subcommittee V on Permanence Properties of A.S.T.M. Committee D-20 on Plastics in connection with the preparation of the present tentative method for evaluating the resistance of plastics to chemicals. The experimental results should be interpreted in accordance with the limitations inherent in the test method, as previously discussed.

MATERIALS AND TEST METHODS

The plastics examined were molded, cast, and paper-base laminated phenol-formaldehyde materials, molded and paper-base laminated urea-formaldehyde materials, casein, cellulose nitrate, cellulose acetate, ethylcellulose, polyvinyl chloride-acetate, polyvinyl butyral, polystyrene in molded and cast forms, polymethyl methacrylate, and cold-molded plastics of the bituminous and phenolic types. The thicknesses and specific gravities of the specimens are listed in Table I. These plastics were obtained from commercial sources and many of them contained the plasticizers ordinarily used in such compositions. Specimens 1 by 3 in. were cut from the sheet materials which were approximately $\frac{1}{8}$ in. thick. The molded specimens were disks 2 in. in diameter and $\frac{1}{8}$ in. thick. The surface areas on these two forms of specimens are approximately equal.

The specimens were placed on edge in test tubes or beakers and totally immersed in approximately 60 ml. of the reagents, which were prepared in accordance with the A.S.T.M. Method D 543.[3] After immersion for 7 days at 25 C., the specimens were removed from the reagents, rinsed in water as prescribed by Method D 543, wiped with a dry cloth,

TABLE II.—PERCENTAGE CHANGES IN WEIGHT AND DIMENSIONS OF PLASTICS IMMERSED FOR 7 DAYS IN CHEMICAL REAGENTS AT 25 C.[a]

	Phenol-Formaldehyde			Urea-Formaldehyde		Vinyl-		Methyl Metha-crylate Resin	Styrene Resin		Cellu-lose Ni-trate	Cellu-lose Ace-tate	Ethyl-Cellulose		Cold-Molded Phenolic	Cold-Molded Bituminous	Casein Plastic
	Lami-nated	Cast	Molded	Molded	Lami-nated	Chlor-ide-Acetate Resin	Butyr-al Resin		Molded	Cast			No. 1	No. 2			
PERCENTAGE CHANGE IN WEIGHT WHEN IMMERSED IN REAGENT FOR 7 DAYS.																	
30% Sulfuric acid	1.9	0.1	0.5	0.2	−0.2	0.04	0.6	0.3	0.01	0.02	0.6	0.8	0.7	0.8	−8.4	−3.7	24.
3% Sulfuric acid	1.6	0.5	2.1	1.0	0.6	0.09	1.8	0.6	0.03	0.05	1.4	3.8	1.6	2.2	−1.6	0.09	48.
10% Nitric acid	6.2	0.4	1.7	1.4	0.4	0.08	3.0	0.5	0.03	0.03	1.4	D	1.3	2.1	−3.0	−0.4	27.
10% Hydrochloric acid	4.8	0.3	1.4	0.3	0.1	0.09	1.5	0.5	0.03	0.05	1.3	11.	1.2	1.2	−8.80	−4.7	34.
5% Acetic acid	1.0	0.6	1.8	1.7	1.1	0.11	3.3	0.7	0.05	0.05	1.6	6.5	1.9	2.9	0.2	1.2	45.
Oleic acid	0.2	0.1	0.2	0.4	0.2	0.02	12.	0.2	0.02	0.01	0.1	0.1	B	B	2.1	1.8	0.6
10% Sodium hydroxide	43.	D	92.	1.0	1.3	0.09	1.2	0.5	0.02	0.04	−1.8	73.	1.1	1.3	D	6.9	D
1% Sodium hydroxide	1.3	6.4	5.0	1.8	1.3	0.09	2.2	0.6	0.03	0.04	0.2	3.3	1.5	2.0	10.	3.0	D
10% Ammonium hydroxide	1.0	4.7	2.6	2.2	1.6	0.16	2.8	0.6	0.10	0.15	8.0	5.8	1.6	3.0	3.0	4.5	105.
2% Sodium carbonate	1.4	0.6	2.0	3.0	0.9	0.08	2.2	0.6	0.04	0.03	1.2	2.6	1.5	1.9	2.0	2.4	43.
10% Sodium chloride	0.5	0.4	1.4	1.2	0.8	0.09	1.5	0.5	0.02	0.04	1.2	2.8	1.2	1.6	1.8	1.6	17.
3% Hydrogen peroxide	1.3	1.0	2.5	1.8	7.1	0.09	2.6	0.7	0.04	0.05	1.4	4.5	1.9	2.1	0.2	0.9	42.
Distilled water	0.8	0.5	2.2	2.0	1.1	0.09	2.5	0.7	0.03	0.03	1.5	4.6	1.7	2.1	1.7	1.4	30.
50% Ethyl alcohol	0.6	1.5	0.9	2.4	0.7	0.07	62.	3.4	0.10	0.11	6.4	25.	38.	36.	1.6	3.2	25.
95% Ethyl alcohol	−0.05	0.04	0.03	−0.3	0.0	0.00	S	11.	0.22	0.14	S	15.	S	S	2.1	2.5	0.4
Acetone	−0.2	0.6	13.	−0.8	−0.6	S	205.	S	S	S	S	S	S	S	3.4	3.9	−3.8
Ethyl acetate	−0.1	−0.3	0.4	−0.4	−0.7	B	B	S	S	S	S	S	S	S	1.6	5.5	2.4
Ethylene chloride	−0.07	−0.1	−0.07	−0.3	−0.1	B	426.	S	S	S	34.	112.	S	S	1.8	12.2	−0.3
Carbon tetrachloride	0.00	0.08	0.03	0.06	0.2	0.14	145.	−24.	S	S	0.20	2.2	S	S	1.9	13.6	−0.2
Toluene	0.2	−0.02	0.04	0.5	0.05	79.	107.	S	S	S	3.4	1.2	S	S	1.6	7.5	−0.1
Gasoline	−0.07	−0.04	−0.01	−0.2	−0.05	−0.01	0.8	0.04	26.	31.	0.03	−0.3	109.	106.	1.4	3.0	−0.1
PERCENTAGE CHANGE IN LENGTH[b] ±0.03 WHEN IMMERSED IN REAGENT FOR 7 DAYS.																	
30% Sulfuric acid	0.05	0.03	0.00	0.10	0.03	−0.03	0.13	0.07	0.00	−0.07	0.10	0.17	0.17	0.14	0.05	−0.10	2.
3% Sulfuric acid	0.15	0.10	0.07	0.45	0.16	0.00	0.23	0.10	0.00	0.02	0.33	1.03	0.33	0.40	0.05	0.30	15.
10% Nitric acid	0.05	0.00	0.07	0.28	0.10	0.07	0.23	0.10	−0.15	−0.02	0.32	A	0.23	0.44	0.00	0.25	9.
10% Hydrochloric acid	0.40	0.07	0.02	0.25	0.10	−0.03	0.16	0.07	0.00	−0.10	0.25	1.2	0.16	0.30	0.10	0.20	5.
5% Acetic acid	0.20	0.10	0.05	0.35	0.13	−0.03	0.26	0.13	0.05	0.03	0.57	1.6	0.26	0.40	0.05	0.40	14.
Oleic acid	0.00	0.03	−0.02	0.10	0.00	−0.03	0.47	0.03	0.05	0.00	0.00	0.04	A	A	0.00	0.10	0.17
10% Sodium hydroxide	3.8	A	0.73	0.25	0.06	−0.10	0.15	0.10	0.05	0.07	0.10	1.1	0.20	0.24	A	0.96	A
1% Sodium hydroxide	0.15	0.42	0.08	0.45	0.03	0.00	0.16	0.17	0.00	−0.07	0.33	1.3	0.30	0.50	1.67	0.35	A
10% Ammonium hydroxide	−0.05	0.23	0.07	0.30	0.07	0.00	0.20	0.10	−0.05	0.00	1.5	−2.4	0.28	0.74	0.05	0.35	32.
2% Sodium carbonate	0.20	0.00	0.10	0.86	0.06	0.02	0.22	0.17	0.00	0.00	0.26	0.73	0.26	0.44	0.25	0.30	14.
10% Sodium chloride	0.25	0.07	0.04	0.35	0.08	−0.10	0.17	0.03	−0.05	0.03	0.27	0.83	0.17	0.32	0.25	0.35	4.
3% Hydrogen peroxide	−0.25	0.07	0.07	0.35	0.24	−0.03	0.22	0.13	−0.10	0.00	0.44	0.99	0.26	0.36	0.00	0.25	17.
Distilled water	0.40	0.09	0.08	0.70	0.11	0.06	0.20	0.16	0.00	0.02	0.42	1.2	0.32	0.48	0.30	0.30	11.

50% Ethyl alcohol	0.15	0.10	0.07	0.45	0.07	0.00	11.	0.20	0.10	0.00	0.90	1.3	4.7	5.7	0.20	0.76	6.
95% Ethyl alcohol	0.20	0.02	0.00	0.10	−0.01	0.00	A	0.43	0.05	0.03	A	−0.20	A	A	0.40	0.40	−0.3
Acetone	0.00	0.11	0.18	−0.15	−0.10	A	A	A	A	A	A	A	A	A	2.2	0.50	−1.2
Ethyl acetate	0.00	−0.03	0.02	−0.05	−0.10	A	A	A	A	A	A	A	A	A	0.25	0.76	−0.6
Ethylene chloride	−0.25	0.02	0.02	−0.15	0.03	A	A	A	A	A	0.66	24.	A	A	0.10	2.3	−0.2
Carbon tetrachloride	0.00	0.04	0.01	0.00	0.05	−0.02	16.	−0.50	A	A	0.02	0.18	A	A	0.00	2.1	−0.1
Toluene	0.05	−0.03	0.02	0.05	0.00	21.	A	A	A	A	0.28	0.38	A	A	0.05	1.8	−0.1
Gasoline	0.00	−0.07	−0.03	0.05	−0.13	−0.03	0.10	0.00	A	A	0.00	0.05	A	A	0.05	0.50	1.7
PERCENTAGE CHANGE IN THICKNESS ±0.8 WHEN IMMERSED IN REAGENT FOR 7 DAYS.																	
30% Sulfuric acid	1.6	−0.8	1.5	0.8	0.4	0.0	0.5	0.0	0.7	0.0	0.0	0.0	0.6	0.3	0.8	1.5	16.
3% Sulfuric acid	1.6	0.8	3.0	0.0	0.0	0.8	0.9	0.0	0.0	−0.3	0.8	2.3	0.5	0.9	0.0	0.0	18.
10% Nitric acid	6.4	0.0	4.6	0.8	1.5	0.0	1.7	0.7	−0.8	−0.3	0.8	A	0.0	0.6	2.3	4.6	20.
10% Hydrochloric acid	6.3	−0.8	2.7	0.8	1.2	0.0	0.4	0.0	0.0	0.0	0.0	11.7	0.3	0.3	1.5	2.4	19.
5% Acetic acid	1.6	0.0	2.2	0.0	0.8	0.0	1.6	0.0	−0.8	0.0	0.8	4.2	0.8	0.9	0.0	0.8	22.
Oleic acid	0.0	0.0	0.4	0.8	0.4	0.0	16.	0.4	0.0	0.6	0.4	0.0	A	A	0.0	0.0	0.8
10% Sodium hydroxide	57.	A	110.	0.0	0.8	0.0	0.4	0.0	0.0	0.0	−2.1	85.	0.6	0.3	A	3.9	A
1% Sodium hydroxide	1.6	14.	8.4	0.0	0.8	0.0	1.3	0.8	0.0	1.3	−1.2	1.9	1.1	0.9	11.5	0.0	A
10% Ammonium hydroxide	1.6	4.7	4.4	0.8	2.3	0.0	2.0	0.0	0.0	0.0	7.8	39.	0.0	1.2	1.5	1.6	46.
2% Sodium carbonate	0.8	0.0	3.8	2.4	−0.4	−0.4	0.4	0.0	0.0	0.0	0.8	1.5	0.3	0.3	4.8	1.5	16.
10% Sodium chloride	0.0	0.8	1.9	0.8	0.0	0.0	0.4	0.8	0.0	0.0	0.0	1.5	0.0	−0.3	0.8	0.0	11.
3% Hydrogen perioxide	1.6	0.8	1.5	0.0	18.	0.0	1.2	0.8	0.0	0.0	0.4	3.0	0.5	0.3	0.8	0.0	15.
Distilled water	0.8	0.0	2.5	1.6	0.0	0.0	1.2	0.0	0.8	−0.3	0.0	2.8	0.3	0.3	1.6	0.0	13.
50% Ethyl alcohol	1.6	1.6	1.4	1.6	0.0	0.0	43.	3.3	0.0	0.0	9.1	43.	31.	30.	−0.8	1.6	14.
95% Ethyl alcohol	0.8	−0.4	2.3	0.8	0.0	0.00	A	15.	0.0	−0.7	A	38.	A	A	2.3	0.8	0.8
Acetone	0.8	4.2	43.	0.0	−1.2	A	A	A	A	A	A	A	A	A	2.3	2.3	−3.
Ethyl acetate	−1.6	0.0	0.8	−1.6	0.0	A	A	A	A	A	A	A	A	A	0.8	1.5	−1.5
Ethylene chloride	0.8	−0.4	0.4	0.0	0.0	A	A	A	A	A	31.	43.	A	A	4.2	9.3	−1.2
Carbon tetrachloride	0.8	0.0	0.4	1.5	0.0	−0.4	91.	−7.4	A	A	0.0	0.0	A	A	0.0	3.8	−1.7
Toluene	−0.8	0.0	1.0	−0.8	0.0	48.	A	A	A	A	7.0	4.9	A	A	−0.8	3.1	−0.8
Gasoline	1.6	−0.8	0.0	0.0	0.0	0.0	3.8	−0.4	A	A	−0.4	0.0	A	A	−0.8	1.6	−0.4

[a] A = Action of reagent on sample made measurements of dimensional changes impractical or impossible; B = Disintegrated by swelling or breaking up; D = Decomposed; S = Dissolved.

[b] For molded specimens, value in length column is change in diameter.

TABLE III.—PERCENTAGE CHANGES IN WEIGHT AND DIMENSIONS OF PLASTICS AFTER IMMERSION FOR 7 DAYS IN CHEMICAL REAGENTS AT 25 C. AND SUBSEQUENT EXPOSURE TO AIR AT 25 C. AND 50 PER CENT RELATIVE HUMIDITY FOR 4 WEEKS.[c]

	Phenol-Formaldehyde			Urea-Formaldehyde		Vinyl		Methyl Methacrylate resin	Styrene Resin		Cellulose Nitrate	Cellulose Acetate	Ethyl-Cellulose		Cold-Molded Phenolic	Cold-Molded Bituminous	Casein Plastic
	Molded	Cast	Laminated	Molded	Laminated	Chloride-Acetate Resin	Butyral Resin		Molded	Cast			No. 1	No. 2			
PERCENTAGE CHANGE FROM ORIGINAL WEIGHT AFTER IMMERSION IN REAGENT FOR 7 DAYS AND SUBSEQUENT EXPOSURE TO AIR AT 77 F. AND 50 PER CENT RELATIVE HUMIDITY FOR 28 DAYS.																	
30% Sulfuric acid	1.2	0.0	0.2	−1.2	−1.0	0.0	0.2	0.0	0.0	0.0	−0.1	−3.4	0.2	0.2	−13.	−7.6	12.
3% Sulfuric acid	0.7	0.1	0.5	−1.3	−0.3	0.0	0.5	0.0	0.0	0.0	0.0	−0.7	0.2	0.3	−4.7	−4.3	8.7
10% Nitric acid	2.6	0.2	0.8	−1.4	−2.2	0.0	0.7	0.2	0.0	0.0	0.3	A	0.3	0.5	−6.3	−5.3	−2.2
10% Hydrochloric acid	1.8	0.1	0.5	−1.9	−3.2	0.0	0.4	0.1	0.0	0.0	0.2	−9.9	0.2	0.3	−13.	−10.	−2.9
5% Acetic acid	0.3	0.1	0.6	0.3	0.4	0.0	0.8	0.2	0.0	0.0	0.2	0.3	0.1	0.5	−1.1	−0.3	−3.7
Oleic acid	0.4	0.1	0.2	0.8	0.3	0.0	12.	0.2	0.0	0.0	−0.2	0.2	A	A	1.9	1.7	0.7
10% Sodium hydroxide	16.	A	30.	0.1	0.0	0.0	0.4	0.1	0.0	0.0	−2.2	−18.	0.1	0.2	A	2.2	A
1% Sodium hydroxide	0.7	−2.5	0.9	0.4	0.2	0.0	0.5	0.1	0.0	0.0	−1.2	−8.3	1.2[a]	0.2	−0.1	0.4	A
10% Ammonium hydroxide	0.0	1.2	0.6	0.9	0.3	0.1	0.7	0.2	0.0	0.0	−1.9	−38.	0.0	0.6	−1.5	−1.7	0.3
2% Sodium carbonate	0.7	0.1[a]	0.5	1.1	0.3	0.0	0.6	0.1	0.0	0.0	−0.2[a]	−2.5	0.0	0.2	0.5	0.2	5.4
10% Sodium chloride	0.3	0.1	0.5	0.2	0.3	0.0	0.5	0.1	0.0	0.0	0.0	−0.2	0.1	0.2	0.1	0.3	3.6
3% Hydrogen peroxide	0.6	0.3[a]	0.5	0.7	−17.	0.0	0.6	0.1	0.0	0.0	0.1	−1.5	−0.1	0.1	−0.8	−1.2	1.5
Distilled water	0.4[a]	0.2	0.6	0.6[a]	0.4	0.0	0.6	0.1	0.0	0.0	0.1	−0.7	0.2	0.1	0.1[a]	0.3[a]	0.3
50% Ethyl alcohol	−0.2	−0.1	0.3	0.9	0.1	0.0	8.7	0.8	0.0	0.0	−1.7	−11.	3.2	2.1	0.0	0.0	3.8
95% Ethyl alcohol	0.1	−0.1	0.1	0.1	0.3	0.0	A	1.9	0.0	0.6	A	−13.	A	A	0.3	0.3	0.3
Acetone	0.0	0.0	4.3	−0.2	0.3	A	A	A	A	A	A	A	A	A	−0.2	−0.3	−1.0
Ethyl acetate	0.1	−0.1	0.4	0.1	0.0	A	A	A	A	A	A	A	A	A	0.4	−0.6	0.2
Ethylene chloride	0.0	0.0	0.0	−0.3	0.1	A	A	A	A	A	−3.8	−12.	A	A	0.2	−1.6	0.1
Carbon tetrachloride	−0.2	0.1	0.2	−0.2	0.2	0.1	33.	−13.	A	A	0.2	−0.3	A	A	0.0	−1.6	0.3
Toluene	−0.1	−0.1	0.2	0.0	0.1	16.	8.2	A	A	A	−1.4	−4.2	A	A	0.0	−1.3	0.2
Gasoline	0.0	0.0	0.1	−0.1	0.0	0.0	0.4	0.1	A	A	−0.6	−0.2	2.1	A	0.1	0.7	0.2
PERCENTAGE CHANGE FROM ORIGINAL LENGTH[b] AFTER IMMERSION IN REAGENT FOR 7 DAYS AND SUBSEQUENT EXPOSURE TO AIR AT 77 F. AND 50 PER CENT RELATIVE HUMIDITY FOR 28 DAYS.																	
30% Sulfuric acid	0.0	0.1	0.0	−0.1	0.0	0.1	0.1	0.0	−0.1	0.1	0.0	−0.2	0.0	0.0	−0.1	−0.3	0.3
3% Sulfuric acid	0.1	0.0	0.0	0.0	0.1	0.1	−0.1	0.0	−0.1	−0.1	−0.1	−0.7	0.0	−0.1	−0.1	0.0	4.2
10% Nitric acid	−0.2	−0.1	0.0	−0.1	0.0	0.0	0.1	0.0	−0.2	0.0	0.0	A	0.0	0.1	−0.2	−0.1	−0.3
10% Hydrochloric acid	−0.1	0.0	0.0	−0.2	0.1	0.0	0.1	0.0	−0.2	−0.1	0.0	0.1	0.0	0.0	−0.1	−0.2	0.2
5% Acetic acid	0.1	0.0	0.0	0.1	0.1	0.0	0.1	0.0	0.1	−0.1	0.1	−0.6	0.0	−0.1	0.0	0.2	2.4
Oleic acid	0.0	0.0	0.0	0.2	0.0	0.0	0.2	0.0	0.0	0.0	−0.1	0.0	A	A	0.0	0.1	0.1
10% Sodium hydroxide	1.9	A	−0.8	0.0	0.1	0.0	0.1	0.0	−0.1	0.0	−0.2	−34.	0.0	0.0	A	0.1	A
1% Sodium hydroxide	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	−1.3	0.1[a]	0.1	0.5	0.1	A
10% Ammonium hydroxide	0.0	0.1	0.0	0.2	0.0	0.0	0.0	0.0	−0.1	0.0	−0.2	−25.	0.0	0.1	0.1	0.1	1.0
2% Sodium carbonate	0.2	0.0[a]	0.0	0.4	0.1	0.0	0.1	0.1	0.1	0.0	−0.1[a]	−1.0	0.0	0.0	0.0	0.0	3.0
10% Sodium chloride	0.1	0.0	0.0	0.1	0.1	0.0	0.0	0.0	0.0	0.0	−0.1	−0.2	0.0	0.0	0.2	0.3	0.4
3% Hydrogen peroxide	−0.1	0.0[a]	0.0	0.3	0.1	0.1	0.0	0.1	−0.1	0.0	0.0	−0.9	0.0	0.0	0.0	−0.2	4.7
Distilled water	0.3[a]	0.0	0.0	0.3[a]	0.1	0.0	−0.1	0.0	0.0	0.0	0.0	−0.7	0.0	−0.1	0.1[a]	−0.1[a]	2.0

50% Ethyl alcohol	0.0	−0.1	0.0	0.1	0.0	0.0	0.1	0.0	0.0	0.0	−2.8	−6.7	−0.2	0.3	0.0	−0.1	0.4
95% Ethyl alcohol	0.1	0.0	0.0	0.1	0.0	0.0	A	0.0	−0.1	0.0	A	−12.	A	A	0.1	0.2	0.0
Acetone	−0.1	0.0	0.0	−0.2	0.0	A	A	A	A	A	A	A	A	A	0.1	−0.3	−0.5
Ethyl acetate	0.1	0.0	0.0	0.2	0.0	A	A	A	A	A	A	A	A	A	0.2	0.0	0.1
Ethylene chloride	−0.1	0.0	0.0	−0.1	0.0	A	A	A	A	A	−13.	6.9	A	A	0.0	0.3	0.0
Carbon tetrachloride	−0.3	0.0	0.0	−0.2	0.0	0.0	−1.5	−0.4	A	A	−0.1	0.0	A	A	−0.2	0.3	0.1
Toluene	−0.1	−0.1	0.0	0.0	0.0	0.9	−3.3	A	A	A	−1.7	−0.9	A	A	−0.1	−0.2	0.0
Gasoline	0.1	0.0	0.0	0.1	−0.1	0.0	0.0	0.0	A	A	−0.1	0.0	8.5	A	0.0	0.4	0.1

PERCENTAGE CHANGE FROM ORIGINAL THICKNESS ±0.8 AFTER IMMERSION IN REAGENT FOR 7 DAYS AND SUBSEQUENT EXPOSURE TO AIR AT 77 F. AND 50 PER CENT RELATIVE HUMIDITY FOR 28 DAYS.

30% Sulfuric acid	0.8	0.8	0.0	0.0	1.3	0.0	0.0	0.0	0.0	0.0	0.0	−3.8	0.0	0.4	0.0	2.3	7.3
3% Sulfuric acid	1.6	0.0	0.0	−0.8	−0.4	0.8	0.4	0.0	0.0	0.0	0.0	0.0	0.5	0.3	−1.6	−3.9	1.7
10% Nitric acid	4.0	0.4	2.3	0.0	1.0	0.0	0.4	0.4	0.0	0.7	0.8	A	0.0	0.3	0.0	1.5	15.
10% Hydrochloric acid	3.9	0.0	0.0	0.8	0.8	0.0	−0.4	0.0	0.0	0.0	0.0	0.7	0.3	0.7	0.8	0.8	11.
5% Acetic acid	0.0	−0.8	0.4	0.0	0.8	0.0	0.8	0.0	0.9	0.0	0.0	1.9	0.3	0.0	−0.8	0.0	1.8
Oleic acid	0.0	0.0	0.0	0.4	0.0	0.0	13.	0.0	0.0	0.3	0.0	0.0	A	A	0.5	2.0	0.8
10% Sodium hydroxide	30.	A	53.	0.0	0.0	0.7	0.0	0.0	0.0	0.0	−2.1	80.	0.6	0.0	A	0.0	A
1% Sodium hydroxide	0.0	2.7	1.9	0.0	0.0	0.0	0.4	0.8	0.8	0.0	−1.3	−6.4	0.6[a]	0.3	1.5	0.0	A
10% Ammonium hydroxide	0.0	0.8	0.0	0.8	0.8	0.0	1.1	0.0	−0.4	0.0	2.5	7.1	0.0	0.0	−0.8	2.3	4.7
2% Sodium carbonate	−0.8	0.0[a]	0.0	0.0	0.0	−0.4	0.0	0.0	0.0	0.0	0.0[a]	0.0	0.6	0.0	0.8	0.0	0.9
10% Sodium chloride	0.0	0.8	0.4	0.0	0.0	0.0	0.8	0.0	0.0	0.0	0.0	0.0	0.0	−0.3	0.8	0.0	2.8
3% Hydrogen peroxide	0.8	0.8[a]	0.0	−1.5	10.	0.0	0.0	0.0	0.0	0.0	0.0	1.5	0.3	0.0	0.0	0.0	5.1
Distilled water	0.0[a]	−0.4	0.8	0.8[a]	−0.4	0.0	0.8	0.0	0.8	0.0	0.0	0.8	0.0	0.0	−4.0[a]	−0.8[a]	0.8
50% Ethyl alcohol	0.8	0.0	0.7	0.0	0.0	0.0	25.	1.1	0.0	0.0	4.2	21.	31.	15.	0.8	1.6	4.8
95% Ethyl alcohol	0.0	−0.4	0.0	0.8	0.0	0.0	A	3.3	0.0	0.0	A	20.	A	A	1.6	0.0	0.0
Acetone	0.8	1.2	8.6	0.0	0.0	A	A	A	A	A	A	A	A	A	0.8	0.8	−2.5
Ethyl acetate	−1.6	0.0	0.4	−1.6	0.0	A	A	A	A	A	A	A	A	A	0.0	−1.5	0.0
Ethylene chloride	0.0	0.0	0.4	−0.8	0.0	A	A	A	A	A	19.	A	A	A	0.0	0.8	−0.8
Carbon tetrachloride	0.8	−0.4	0.3	0.8	0.0	−0.4	23.	−2.1	A	A	0.0	−0.8	A	A	0.0	−0.8	0.0
Toluene	0.0	0.0	0.4	−0.8	0.0	24.	19.	A	A	A	−2.5	−0.4	A	A	0.0	1.5	0.0
Gasoline	−0.8	0.8	0.0	0.0	0.0	0.0	0.0	0.0	A	A	−1.2	−0.4	33.	A	−2.3	−0.8	0.0

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[a] These values were obtained 7 days after removal from the reagent.

[b] For molded specimens, value in length column is change in diameter.

[c] A = Action of reagent on sample made measurements of dimensional changes impractical or impossible.

and weighed without delay. The diameter or length and the thickness were measured immediately after weighing. The specimens were left in a room conditioned at 25 C. and 50 per cent relative humidity and the weights and dimensions were measured 1 week and 4 weeks after removal from the reagent.

An analytical chemical balance was employed for determining the weights of the specimens. Dimensions were measured with dial gages reading to 0.001 in. and having approximately 400 g. per sq. cm. pressure on the foot. All percentage changes in weights and dimensions are calculated on the basis of the weights and dimensions of the specimens as received.

TEST RESULTS AND DISCUSSION

The changes produced in the weights and dimensions of the plastics specimens by immersion in the reagents for 7 days at 25 C. are shown in Table II. It is possible by examination of the vertical columns to obtain a general picture of the resistance of a given plastic to the various types of chemicals. Likewise, by following across the table horizontally one can see the relative resistances of the various types of plastics to a given reagent. Thus, the top line of figures indicates that styrene and vinyl chloride-acetate resins are outstanding in their resistance to 30 per cent sulfuric acid. Looking down the first column of figures, it is readily apparent that the phenolic molding composition containing wood flour is markedly attacked by 10 per cent sodium hydroxide solution. Similar observations for the other chemicals and plastics can be readily made from the data in Table II. For those reagents involving aqueous solutions it is helpful to compare the values observed for immersion in water alone with those measured after immersion in the solutions.

The results obtained in gasoline cannot be taken as representative of the effect of aliphatic hydrocarbons on the plastics, as it is probable that some naphthenes and aromatic hydrocarbons were also present. For this reason, the subcommittee which prepared this method has under consideration a proposal to substitute a typical aliphatic hydrocarbon for gasoline in this test. Considerable variation in the action on some plastics of the three gasolines used in the tests reported in this paper was noted.

In examining the values for changes in thickness, it is important to remember that the measurements were made only to the nearest 0.001 in. and that changes of less than about 1 per cent are not significant.

Data relating to the recovery of the original weights and dimensions by the specimens of plastics after removal from the chemical reagents are presented in Table III. The values indicate the percentage changes from the original condition of the specimens after they had been removed from the reagents and exposed for 4 weeks at 25 C. and 50 per cent relative humidity. In many instances negative values are observed which can be attributed to the removal of a portion of the plastic composition by the chemical reagent. This is particularly pronounced for cellulose acetate plastic, from which plasticizers were apparently leached.

Comparison of the data in Tables II and III will reveal that for some materials the absorption of the chemical reagent over the 7-day period was nearly balanced by the removal of soluble constituents from the plastic. Thus, for cold-molded, phenolic plastic the gain in weight during 7 days immersion in a 5 per cent solution of acetic acid was only 0.2 per cent, but the weight of the plastic 28 days after removal from the

TABLE IV.—EFFECT ON PLASTICS OF IMMERSION FOR 7 DAYS IN CHEMICAL REAGENTS AT 25 C.

	Phenol-Formaldehyde			Urea-Formaldehyde		Vinyl		Methyl Methacrylate Resin	Styrene Resin		Cellulose Nitrate	Cellulose Acetate	Ethyl-Cellulose		Cold-Molded Phenolic	Cold-Molded Bituminous	Casein Plastic
	Molded	Cast	Laminated	Molded	Laminated	Chloride-Acetate Resin	Butyral Resin		Molded	Cast			No. 1	No. 2			
30% Sulfuric acid	Surface roughened	None	Edges swollen	Surface roughened	Surface attacked	None	None	None	None	None	None	Crazed; softened	None	None	None	None	Rubbery
3% Sulfuric acid	Surface roughened	None	Edges swollen	Surface roughened	Surface attacked	None	Cloudy	None	None	None	None	Swollen	None	None	None	None	Swollen; rubbery
10% Nitric acid	Surface roughened	None	Edges swollen	Surface roughened	Delaminated	None	Cloudy	None	None	None	None	Decomposed	None	None	None	None	Swollen; cracked
10% Hydrochloric acid	Surface roughened	None	Edges swollen	Surface roughened	Delaminated	None	Cloudy	None	None	None	None	Decomposed	None	None	Cracked on drying	Cracked on drying	Swollen; cracked
5% Acetic acid	None	None	Edges swollen	None	None	None	Cloudy	None	None	None	None	Swollen	None	None	None	None	Rubbery; split
Oleic acid	None	None	None	None	None	None	Tacky	None	None	None	None	None	Decomposed	Decomposed	None	None	None
10% Sodium hydroxide	Decomposed	Decomposed	Delaminated	None	Surface attacked	None	None	None	None	None	Crazed	Decomposed	None	None	Decomposed	None	Decomposed
1% Sodium hydroxide	Surface roughened	Decomposed	Edges swollen	None	None	None	Slightly cloudy	None	None	None	Crazed	Surface attacked	None	None	Decomposed	None	Broken up
10% Ammonium hydroxide	Surface dulled	Discolored	Discolored; edges swollen	None	None	None	Opaque	None	Discolored	None	Crazed; discolored	Opaque; soft	None	Dye attacked	None	Surface attacked	Swollen; split
2% Sodium carbonate	None	Discolored	Discolored	None	None	None	Slightly cloudy	None	None	None	None	Swollen	None	None	None	None	Swollen; rubbery
10% Sodium chloride	None	None	Edges swollen	None	None	None	None	None	None	None	None	None	None	None	None	None	None
3% Hydrogen peroxide	None	Discolored	None	Surface dulled	Delaminated	None	Cloudy	None	None	None	None	None	None	None	None	None	Swollen; rubbery
Distilled water	None	None	None	None	None	None	Cloudy	None	None	None	None	None	None	None	None	None	Swollen; rubbery
50% Ethyl alcohol	None	None	None	None	None	None	Swollen; rubbery	Slightly swollen	None	None	None	Partly dissolved	Swollen; cracked	Swollen; cracked	None	None	Swollen; rubbery
95% Ethyl alcohol	None	None	None	None	None	None	Dissolved	Surface attacked	None	None	Dissolved	Partly dissolved	Dissolved	Dissolved	None	None	None
Acetone	None	Softened	Blistered	None	None	Dissolved	Swollen; opaque	Dissolved	Dissolved	Dissolved	Dissolved	Dissolved	Dissolved	Dissolved	None	Broke in half	None
Ethyl acetate	None	None	None	None	None	Decomposed	Decomposed	Dissolved	Dissolved	Dissolved	Dissolved	Dissolved	Dissolved	Dissolved	None	None	None
Ethylene chloride	None	None	None	None	None	Dissolved	Decomposed	Dissolved	Dissolved	Dissolved	Partly dissolved	Soft; swollen	Dissolved	Dissolved	None	Cracked	None
Carbon tetrachloride	None	None	None	None	None	None	Swollen; rubbery	Surface attacked	Dissolved	Dissolved	None	None	Dissolved	Dissolved	None	Cracked	None
Toluene	None	None	None	None	None	Soft; rubbery	Swollen; rubbery	Dissolved	Dissolved	Dissolved	Partly dissolved	None	Dissolved	Dissolved	None	Cracked	None
Gasoline	None	None	None	None	None	None	None	None	Partly dissolved	Tacky; bubbled	None	None	Swollen; cracked	Swollen; cracked	None	None	None

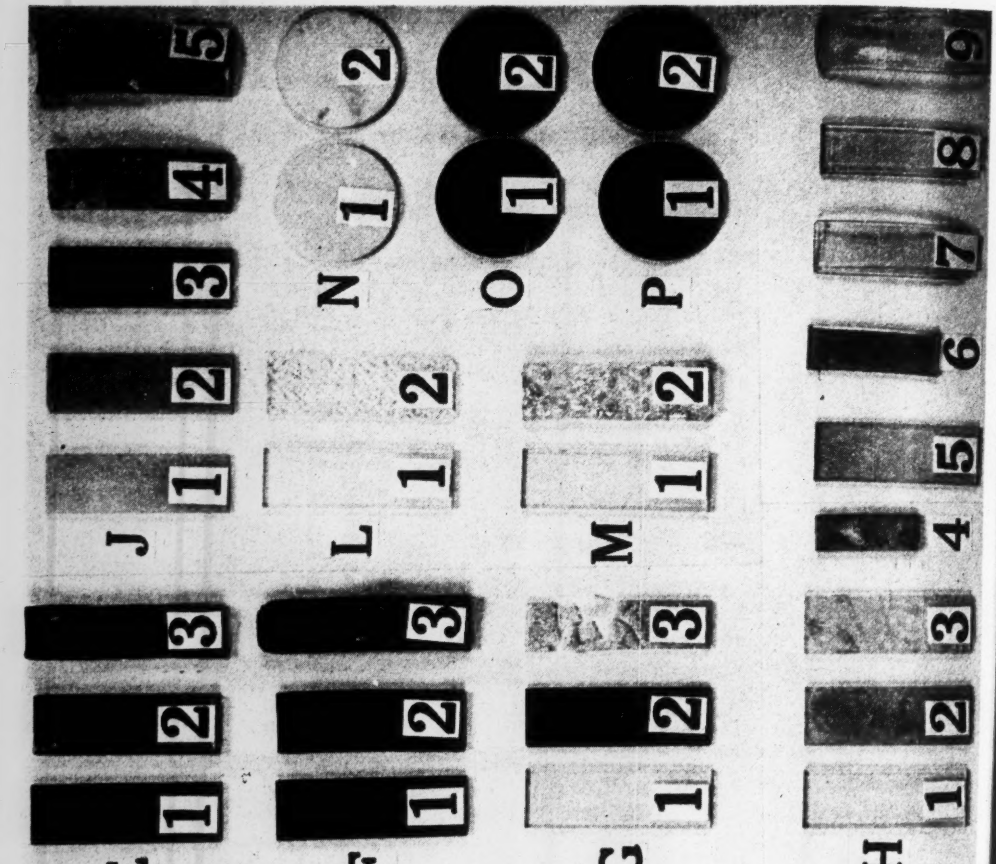
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FIG. 1.—Appearance of

FIG. 1.—Appearance of Various Plastics After Immersion for 7 days in Chemical Reagents at 25 C.

A—Phenol-formaldehyde cast
  1. Original
  2. 10% Nitric acid
  3. 1% Sodium hydroxide
  4. 2% Sodium carbonate

B—Phenol-formaldehyde laminated
  1. Original
  2. 10% Hydrochloric acid
  3. 10% Sodium hydroxide
  4. 1% Sodium hydroxide
  5. Acetone

C—Urea-formaldehyde laminated
  1. Original
  2. 30% Sulfuric acid
  3. 10% Sodium hydroxide
  4. 3% Hydrogen peroxide

D—Vinyl butyral resin
  1. Original
  2. Oleic acid
  3. Acetone
  4. Ethylene chloride
  5. Carbon tetrachloride

E—Ethylcellulose No. 1
  1. Original
  2. 50% Ethyl alcohol
  3. Gasoline

F—Ethylcellulose No. 2
  1. Original
  2. 50% Ethyl alcohol
  3. Gasoline

G—Cellulose nitrate
  1. Original
  2. 10% Ammonia
  3. Ethylene chloride

H—Cellulose acetate
  1. Original
  2. 30% Sulfuric acid
  3. 10% Hydrochloric acid
  4. 10% Sodium hydroxide
  5. 1% Sodium hydroxide
  6. 10% Ammonia
  7. 50% Ethyl alcohol
  8. 95% Ethyl alcohol
  9. Ethylene chloride

J—Casein
  1. Original
  2. 30% Sulfuric acid
  3. 10% Nitric acid
  4. 10% Hydrochloric acid
  5. 10% Ammonia

L—Methyl methacrylate
  1. Original
  2. Carbon tetrachloride

M—Polystyrene
  1. Original
  2. Gasoline

N—Urea-formaldehyde molded
  1. Original
  2. 10% Nitric acid

O—Cold-molded phenolic
  1. Original
  2. 1% Sodium hydroxide

P—Cold-molded bituminous
  1. Original
  2. Ethylene chloride

reagent was 1.1 per cent less than the original weight. Likewise, for cold-molded bituminous plastic the 7-day gain in weight in 3 per cent sulfuric acid was only 0.09 per cent, but the weight 28 days after removal from the reagent was 4.3 per cent under the original weight. Hence, it is obvious that unless the weight is determined after reconditioning in air, a false impression may be gained regarding the effect of chemicals on some plastics.

Information regarding the physical appearance of the plastics after immersion in the various chemicals is presented in Table IV. Many evidences of attack not revealed by changes in weight or dimensions are listed in this table. Various specimens illustrating the effects produced by the reagents were selected for photographing. These are shown in Fig. 1. Edge discoloration of the paper-base laminated phenolic plastic, delamination of the urea-formaldehyde specimen, warping of the vinyl butyral and casein materials, and pronounced shrinkage of cellulose acetate plastic when dried after immersion in certain reagents are among the phenomena illustrated.

A final word of caution may be desirable in order to avoid misinterpretation of the information contained in Tables II, III, and IV. Effects which do not become prominent in a 7-day immersion period or a subsequent 4-week reconditioning period may under conditions of service bring about relatively rapid deterioration of the plastics. This is particularly true for the plastics immersed in strong acids. Thus, although cellulose nitrate plastic is indicated as unaffected by the 7-day immersion in sulfuric, hydrochloric, and nitric acids, the presence of the free acid in this plastic will accelerate its decomposition. Another precaution to be observed is in using the data for guidance in applications involving contact successively with various chemical reagents. Cellulose nitrate plastic is relatively unchanged in appearance by a 7-day contact with 10 per cent nitric acid and carbon tetrachloride, respectively. However, if the sample of cellulose nitrate which has been dried in air for 4 weeks after immersion in 10 per cent nitric acid is kept for 7 days in carbon tetrachloride, it swells, develops blisters, and becomes markedly discolored. Although this is an extreme example, it illustrates the difficulties that may be encountered unless adequate tests are made with plastics under the actual conditions to which they will be subjected in service.

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DISCUSSION

MR. J. H. ADAMS.[1]—The plastics industry is indebted to the authors for calling attention to the need for chemical resistance tests. We have given much thought to measuring mechanical and electrical properties, and so forth, but very little, relatively, to chemical properties. There undoubtedly are big markets where chemical resistance is a requisite, which may be had for plastics, if we know enough about what they will do. This test is a big step towards determining which plastics are entirely unsuited for such uses. I think, however, we need to go much beyond that. After we have eliminated those that are obviously unsuited, we still need to know whether those that are left are good enough, and that is a much larger problem.

This matter of chemical resistance is a very large problem indeed. We have all these various reagents, and various materials to consider; we have questions of temperature of exposure; we have questions of alternate wet and dry exposures; we have questions of how the plastic is fabricated, all of which enter in. It is quite a problem to get straightened out, and I should like to take this opportunity to direct your thought toward establishing a few fundamentals in this big sea of needed information. It seems to me the base line we need is to determine which reagents will be entirely destructive to plastics—that is, what sort of reagents, if exposure were continued, would in time destroy the plastic. We need to identify and classify those which may not be continuously destructive, but may come to some maximum, and to know about how long it is going to take, about how far and along what lines they may go in their destructive action.

These are facts which we need for a base line in order that we may develop accelerated tests. Unless we know about what will happen with long time, we will not know what we are trying to measure in a short time.

What I am trying to stress is that work should be directed toward finding out which sort of reagents will go on destroying the plastic indefinitely, and which will reach a maximum, and then I think work should be directed toward finding out what observations are required to judge attack, and how various observations may be related. For example, what is the significance of weight change? After all, we are not particularly concerned whether a plastic object gets heavier or lighter. We are concerned with whether it is still serviceable, whether it is still as strong, whether it retains other properties that we may want.

In regard to weight change, I think we should be particularly conscious of the need for starting with some fixed base of moisture content, because plastics do absorb and lose moisture, and weight changes are affected by initial moisture contents.

Then there is the question of appearance. Appearance is sometimes taken as very indicative. Well, appearances can be very deceiving, unless it is appearance that we are concerned with.

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Then there is the matter of strength or other physical properties. In strength tests, drying out may be quite a factor. That is, the material when it is wet may not have lost much strength, but depending upon how it is dried out, it may lose considerable strength in its relation to weight. Whether it is dependent on weight changes is a very important thing to establish.

I think if everyone working on this problem would try to picture it in this way of establishing fundamental facts about chemical resistance, we could arrive at very good ways of evaluating plastics.

MR. S. J. ROSCH.[2]—I was struck by what Mr. Adams said. If I understood Mr. Adams correctly, he said that before we set up accelerated aging tests, we should get an idea of what is going to happen to the material in service over a long period of time. How are you going to do that without waiting say, ten or fifteen years? How are you going to set up an end point? We must set up accelerated tests, so as to try to anticipate what is going to happen at the end of ten or fifteen years.

MR. ADAMS.—That is very true. But I am trying to take the long-time view of this. That is, let us not go on making accelerated tests without planning to find out what the long-time answer is going to be. Let us keep in mind, that if temporarily we have to make accelerated tests, let us at the same time see that long-time tests are started, so that eventually we will know whether they do agree. Somehow the knowledge has to be gotten as quickly as it can as to what long-time effects are going to be.

MR. DONALD L. GIBB.[3]—It frequently has been said that the tests approved by the A.S.T.M. are for referee purposes, implying thereby that they do not have practical application. This, however, is not true, since the referee test is, of itself, a practical thing. The proposed test for chemical resistance has definite practical significance in that it tends to narrow down the field which an investigator need cover in determining whether a given plastic is suitable for a proposed application.

It is impossible, in outlining a test of this kind, to cover all the contacts into which a plastic might come in service. For example, it is easily conceivable that one of the prime requisites for a plastic steering wheel might be resistance to perspiration. In this case, the chemical is not of a specific nature or definite concentration, since it is highly probable that the perspiration of one individual is not identical to that of another. In the case of plastic dentures, the action of the mouth fluids in many cases has a deleterious effect. Unfortunately, however, the conditions in one mouth cannot be taken as representative of those in all mouths; but this truly is a case in which chemical resistance is of prime importance.

A type of chemical resistance which has not been considered in this test, and might be of interest, is the resistance of plastics to lacquers or of plastics to plastics, since it is a well-known fact that migration of plasticizers and color frequently occur from one of these materials to the other.

Closely allied to the resistance of plastics to chemicals is the resistance of chemicals to plastics. It is conceivable, for example, that a plastic which is apparently unattacked by sulfuric acid of battery strength may so affect the battery acid as to impair the efficiency of the unit. This is the type of thing that could not be determined in the average laboratory by the type of chemical resistance test which has been proposed, the use being so specifically technical that probably only the battery manufacturer himself would be in a position to measure accurately the effect of such contact. In a similar type of application the effect of the plastics on the contact material might easily be confused by its ability to absorb the material in which it was suspended. For example, plastics used on plating hooks in electroplating baths might transfer from one bath to another the chemicals with which they were last in contact, and so poison the bath as to render it ineffective.

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[3] Technical Representative, The Dow Chemical Co., Midland, Mich.

It is unfortunate that tests cannot be devised which will immediately determine the effect of given chemicals on any plastic after prolonged exposure, say 8 to 10-yr. contact; but the test which has already been proposed does provide a practical method for comparison and elimination and makes it possible for a user to determine, in a preliminary way, the probable suitability of available materials.

MR. J. C. PITZER.[4]—Mr. Kline and his coworkers have performed a commendable piece of work in this initial presentation on the chemical resistance of plastics. I should like to give just one word of caution to that person who may consider plastics in chemical applications, and that is this:

Plastics does not refer to a definite composition of material, and the producer of plastics may very readily modify a product to the extent that it may not in the initial trial be satisfactory, but with certain modifications be entirely satisfactory for the application.

To that end, I want to point out one example—phenolic laminated material made with an asbestos filler. With an ordinary asbestos cloth filler, the product is very unsatisfactory in sulfuric acid applications; yet the same phenolic resin impregnated on suitable cellulose paper has been found to be entirely satisfactory.

If the first trial you perform with a plastic is not satisfactory, go to the producer, tell him the results of your first trials and ask him to submit another material.

MR. G. M. KLINE.[5]—I want to thank these gentlemen for their comments. I think they have expanded on the thoughts that were stressed in the paper, as to the need of caution in interpreting the results of chemical resistance tests.

In conclusion, I am reminded of a quotation from Shakespeare that might be considered in developing these test methods. Slightly modified, it runs:

"... that but this 'test method'
Might be the be-all and the
end-all here."

But unfortunately, I do not think we will ever reach that stage.

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[5] Chief, Organic Plastics Section, National Bureau of Standards, Washington, D. C.

SYMPOSIUM ON PROBLEMS AND PRACTICE IN DETERMINING STEAM PURITY BY CONDUCTIVITY METHODS

INTRODUCTION

By R. E. Hall[1]

The comprehensive array of purifiers, baffles, washers, and separators, which has been applied to the separation of boiler water from steam, and the intensive efforts which have wrought their development, convincingly manifest the tremendous importance which attaches to purity of steam—rightly so, too, since the efficiency of the turbine is directly dependent thereon. To establish the criteria by which these developments could be evaluated, methods of measuring the purity of steam necessarily have undergone thorough revision and refinement. In this process, the determination of steam purity by the electrical conductivity method has quite generally replaced the older procedures.

Why this is the case becomes readily apparent when the electrical conductivity and the throttling-calorimeter method are compared.

The throttling calorimeter commonly used is essentially a device for measuring the Joule-Thomson coefficient $\left(\frac{\delta T}{\delta p}\right)_h$ of commercial steam over the pressure range $p_{\text{saturation}} - p_{\text{atmospheric}}$. By comparison of the data so secured with those on dry steam which comprise our steam tables, a result is derived which represents the percentage of moisture in the commercial steam.

As Kleinschmidt (**31**)[2] has pointed out, the Joule-Thomson coefficient is a very sensitive means of computing steam properties, and even where large differences exist between individual sets of determinations of the coefficient and between various empirical formulations thereof, the computed values of enthalpy, etc., appearing in steam tables of various authorship, differ in much smaller proportion. When, however, the quality of steam is computed from a comparison of the data from the throttling calorimeter with the steam tables or Mollier Diagram, the computations of the compiler thereof are in effect reversed. Hence, there will be included in the result all the error resulting from not following the elaborate precautions taken in the compilation of the tables, among them, elimination of such sources of error as back pressure in the calorimeter, kinetic energy in the steam passing over the thermometer bulb, thermometer calibration, and even loss of heat from the calorimeter body and connecting piping. The unavoidable lack of precision in such procedure is an understandable reason why for decades "commercially dry steam" was considered to contain 1 to 2 per cent moisture when in fact it may have been dry or nearly so. In fact, all calorimetric determinations which indicate a practically unvarying percentage of moisture of more than a few tenths, infer strongly that the apparent moisture content is really a measure of the inaccuracy of the calorimetric method. Further, in view of the fact that the same data compared with different modern steam tables yield quality figures differing by tenths of a per cent, what hope is there that the test engineer in a boiler plant can determine the magnitude of the Joule-Thomson effect with sufficient accuracy to make his results worth while? Only one technique will yield satisfactory results; the calorimeter must be standardized *in situ* by operating it and the boiler under such conditions that it is known the steam entering it is dry. This is the method recommended by The American Society of Mechanical Engineers in its Power Test Code, Series 1929, Part II. Thus calibrated, a calorimeter should yield data which can be relied upon to indicate the quality of the steam *entering the sampling nozzle* to within 0.1 per cent or so. Without such calibration, results are meaningless.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

Furthermore, as Markson and Olsen point out in their recent article on "The Flashing Calorimeter," (36) "The throttling calorimeter becomes theoretically useless at pressures in excess of 1800 lb. absolute. As a practical matter, the throttling calorimeter decreases in usefulness once the pressure of 575 lb. is passed, because of decreasing enthalpy of the vapor."

The calorimeter determines moisture in steam; only indirectly, either by analysis of the condensed steam or by reference to the composition of the boiler water, does it give the value of the solid constituents in the steam. More recently, the tendency—perhaps the rule—is to place the emphasis on the cleanliness of the steam. Just as soon as the criterion of steam quality is made, not percentage of moisture, but parts per million of foreign constituents, the direct estimation of the latter by the electrical conductivity or other method must supersede in value its indirect estimation from the moisture percentages of the calorimeter. The accuracy and adaptability of the electrical conductivity method, combined with its rapidity of measurement and the ease of automatically recording the results continuously over considerable periods of time, have given the method well-justified popularity and widespread use. Thus, because of its lack of precision and its indirect measurement only of solid constituents, the calorimeter has become outmoded.

For certainty that the accuracy of the electrical conductivity method will be 1 ppm. or less of contaminating solids, a number of factors must be given careful consideration. While further improvement and development will undoubtedly ensue, this symposium has for its purpose the summation and critical evaluation of those principles which are pertinent to the method, discussion of which has until now been scattered through the reports of a considerable number of investigations.

On one point, that of the derivation of the sample from the flow of steam in its main, the A.S.M.E. method as described in Power Test Code, Series 1929, Part II, has stood the test of time. But as soon as any condensation begins, accuracy demands that the composition of any sampling connections and containers in contact with the steam or its condensate shall be carefully chosen in order that dissolution thereof shall not change the resistivity or conductivity of the sample under consideration. This phase is discussed by Belyea and Moody.[3]

The quest in the conductivity measurement is to determine the solids in the steam. It is, therefore, essential that the effect of any other components such as gases which dissolve in its condensate and increase its conductivity, shall either be removed or corrected for. The subject of correction for these gases has received considerable attention, and represents one main line of attack on this problem. This phase is discussed in two papers, the first by Whirl and Lower,[4] and the second, by McKinney.[5]

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[3] See p. 1264.

It is highly desirable, however, that the direct reading of the conductivity meter shall represent without correction the conductivity contributed by the solids only. This is especially true where the conductivity measurements are recorded on charts hour after hour. Great effort is being made to contrive means whereby contaminating gases will be removed from the sample automatically before its entry into the conductivity cell, whereupon the reading of the conductivity meter will represent directly the amount of contamination by solids in the sample. Quite different methods of approach to this problem are presented in the papers by Place[6] and Mumford,[7] respectively.

Finally, with wide scope of conductivity equipment to choose from, judgment must be used in the selection of an instrument appropriate for an intended application with emphasis, but not overemphasis, upon refinement of measurement, lest the worship of precision dwarf the attention given to the interpretation and meaning behind the measurement. Kaufman presents this phase of the symposium program.[8]

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[4] See p. 1277.
[5] See p. 1285.
[6] See p. 1302.
[7] See p. 1314.
[8] See p. 1326.

THE SAMPLING OF STEAM AND BOILER WATER

By A. R. Belyea[1] and A. H. Moody[1]

Synopsis

The principles of representative sampling as applied to the sampling of steam and boiler water are reviewed. The extension of steam temperatures and pressures to higher values in modern steam boiler and turbine design imposes stricter requirements in the sampling of steam and boiler water, especially as regards accuracy of sampling and freedom from traces of contamination from sampling equipment. Sampling lines, cooling coils, and sample containers must preserve the integrity of the sample by suitable choice of materials of construction. Metals and alloys for this purpose are discussed from the standpoint of corrosion resistance in the presence of the chemical impurities in the steam, boiler waters, and cooling water.

Modern high pressure boilers are characterized by complex design, small ratio of boiler water capacity to rate of steam release, and rapid steaming rates, resulting in sensitiveness, as regards carry-over, to small changes in concentrations of chemical constituents in the boiler water. Modern turbines are characterized by reduced size per unit of electrical output and are sensitive, as regards blade deposits, to very small concentrations of soluble and insoluble solids in the steam. These developments have brought about refinements in testing methods to meet demands for closer limits of operation and more accurate control. The growing value of the use of hydrogen ion concentration and conductivity measurements has emphasized the need for careful sampling practices to insure the maximum accuracy obtainable. The importance of a properly representative sample, free from contamination, cannot be overstressed, since all subsequent analytical determinations and their interpretations can be no better than the sample itself.

In the sampling of steam and boiler water, obtaining a properly representative sample implies:

1. Proper choice of sampling locations.
2. Withdrawal of sample at the chosen locations in a proper manner.
3. Bringing the sample, without contamination or loss of integrity, from elevated temperature and pressure to atmospheric pressure and a temperature suitable for analysis.
4. Collecting sufficient volume of sample for ensuing analytical determinations.
5. Collecting samples with sufficient frequency to accurately gage changing conditions within the container from which the sample is taken.
6. Storing the sample prior to analysis in such a manner as will safeguard against change in the particular properties to be determined.

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CHOICE OF SAMPLING LOCATIONS

The point of sampling must be chosen with extreme care and with a clear understanding of the purpose of the sample and the use to be made of the test results. Selection of sampling points should not be undertaken without a thorough knowledge of the operation and functions of the equipment from which the sample is to be taken. In modern boilers of high capacity and high pressure the rapid throughput of feedwater and the complicated circulation during operation make it advisable to conduct a survey of the boiler by taking samples at many points in order to determine the proper point or points to obtain a sample that will furnish the desired information. Such a survey should be of sufficient duration to adequately average the effect of load changes (**16**).[2]

For example, a perforated pipe inserted lengthwise in a boiler drum or collecting header for collecting an "average" sample will often draw steam preferentially from the perforations nearest the header or drum shell. Baker (**3**), in a study of mechanical purification of steam within the steam release drum of a 1400-psi. boiler, found the steam to be of varying degrees of purity along the length of the drum; eight sampling points were used in this case.

OBTAINING A REPRESENTATIVE SAMPLE OF STEAM

The sampling of steam may be considered as the sampling of a gaseous fluid containing liquid or solid particles or both. A representative sample of such a fluid must contain the same ratio of number and size of particles to fluid as exists in the container or main that is being sampled. Hence when sampling steam in motion, as in mains, it is necessary to extract the sample at the same linear velocity as the steam passing the sampling point (**27**). It is preferable to insert the sampling nozzle into a vertical straight run of pipe some distance from any bend or fitting in order to minimize the effects of possible stratification. If the stream to be sampled is not uniform due to stratification, more than one sampling point may be advisable. A.S.M.E. Power Test Codes (**13**) give the principles of application and location of sampling nozzles in connection with steam calorimetry. Their standard nozzle has been criticized and alternative types suggested (**43, 39, 62**).

OBTAINING A REPRESENTATIVE SAMPLE OF BOILER WATER

Developments in the modern steam boiler have placed more stringent requirements on sampling procedure for boiler waters. General experience has been that with higher rates of evaporation the concentrations of salines and especially of residual treating chemicals must be carefully controlled within narrow limits to avoid carry-over. As in the case of steam sampling, the withdrawal of the boiler water sample from the selected point or points must be performed in such a manner as to insure the same ratio of solids to liquid in the sample as in the fluid being sampled. Sufficient sample water must be discarded or discharged overboard at the start of collection so that any stagnant water in the lines or at the point of sampling is disposed of prior to sample collection. This is especially necessary if appreciable quantities of suspended or insoluble solids are present which may settle out at or near the sampling point. It is also advisable to do likewise in the case of new sampling nozzles and lines, as many of the commonly used metals develop protective interior surface coatings on initial contact with boiler water, and this reaction should be completed prior to actual sample collection where extreme accuracy is required.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

A metal filter may be inserted at the point of sampling if it is desired to remove the insoluble solids from the boiler water before cooling. This may be necessary to avoid reaction between insoluble solids and the boiler water while the sample is being cooled and collected or to avoid atmospheric contamination during laboratory filtration by ordinary methods. Collins and Schroeder (6) indicate the extent of change in the character of boiler water samples caused by atmospheric contamination and describe a copper screen filter for use at the boiler sampling point.

A.S.T.M. Tentative Methods of Sampling Plant or Confined Waters for Industrial Uses (D 510 – 40 T)[3] should be consulted for accepted sampling practice. General directions for sampling water in a stationary boiler are given in American Public Health Association standards (55). A comprehensive discussion of the factors involved is given in the reports of members of the Joint Research Committee on Boiler Feedwater Studies sponsored by the A.S.T.M. and other societies (49, 50, 6, 42).

SAMPLING LINES

The choice of materials for construction of sampling lines must take into account the pressures and temperatures involved, to avoid structural failure. Except for strongly acid or alkaline waters, chemical considerations are not so pronounced, since in most instances time of contact of the sample and sampling line can be minimized through judicial choice of rate of flow and diameter of line. On the other hand, the sampling lines will operate generally at a continuously higher temperature than those parts of the sampling equipment in contact with cooling water, so that any progressive chemical reaction will be more rapid due to the higher temperature. As a generalization, the use of the same material for sampling lines as is used for the container from which the sample is being withdrawn is permissible, provided contact times are short. In many instances the metal or alloy chosen for the sampling line is based on the choice for cooling coil construction.

COOLING COILS

In bringing the sample to pressure and temperature conditions suitable for storage and analysis, the prevention of contamination is of major importance where conductivity measurements are to follow, as relatively small concentrations of dissolved gases or metallic ions may introduce appreciable errors in the determination. Hence the corrosion resistance of metals and alloys suitable for construction of sampling lines and cooling coils is a major consideration in modern sampling practice.

The cooling coil must handle the quantity of sample decided to be necessary; reduce the temperature of the sample, generally to room temperature; and discharge the sample at a lower pressure, usually atmospheric. It is not within the scope of this paper to present any standard design of cooling coil or to specify the physical dimensions of such cooler for a given quantity of heat absorption. The amount of heat-absorbing surface required will vary with the temperature and velocity of the sample water or condensate and the temperature and rate of flow of the cooling water, as well as with the design of the coil.

In considering corrosion resistance for

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[3] 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 523.

cooling coil service, not only the nature of the condensed steam or boiler water must be considered but the nature of the cooling water as well; the latter may be the controlling factor governing the selection of materials. A common design of cooling coil consists of two tubes, one within the other (18). The cooling water enters the outer tube at the bottom and leaves at the top, completely surrounding the inner tube at all points. The hot test sample enters the top of the inner tube and leaves at the bottom. While there are slight modifications of the foregoing design between coils supplied by manufacturers, such as the use of tube and shell instead of two tubes, the principle of counter current flow is usually employed.

On the sample side of the coils we may have the following in contact with the metal:

1. Boiler waters, containing small amounts of oxygen, ammonia, and other dissolved gases; insoluble carbonates, sulfates, and phosphates of calcium, iron, and magnesium; soluble chloride, carbonate, bicarbonate, sulfate, sulfite, silicates, hydroxide, and phosphates of sodium; tannin, starch, and a variety of other chemical compounds.

2. Condensed steam, containing small amounts of carbon dioxide, oxygen, ammonia, and other gases; and liquids and solids from boiler water, as enumerated in the preceding paragraph.

The boiler waters will almost always be alkaline, usually above pH 9.0, while the steam condensate may be acid due to dissolved carbon dioxide, or alkaline due to boiler water carry-over. Temperatures to 950 F. and pressures to 2300 psi. may be encountered; pressures to 3200 psi. are in prospect.

On the cooling side of the coils the following types of water may be met:

1. Good city water, or good condensate, containing but small amounts of carbon dioxide and oxygen.

2. Poor city water, containing temporary hardness due to carbonates and bicarbonates of calcium and magnesium.

3. Industrial fresh water from wells, streams, and similar sources, containing carbon dioxide dissolved or chemically combined, dissolved oxygen, hardness, sand, algae, and sewage. This type of water may be acid due to industrial wastes or organic acids or may be high in ammonia and proteins.

4. Brackish water, such as tide water, consisting of fresh and salt water in varying proportions and corresponding proportions of impurities of industrial fresh water and salt water.

5. Salt water, containing dissolved gases such as oxygen; chlorides, sulfates, carbonates, and bicarbonates of sodium, potassium, calcium, and magnesium; algae; and sewage.

The temperature of the cooling water may be from 32 F. upwards. Pressures normally will not exceed 100 psi.

In considering metals for the fabrication of cooling coils to operate under the foregoing general conditions, a wide variety of useful corrosion-resistant metals and alloys is available for choice. The problem is to select the material that best meets the particular set of conditions confronting the user with regard to corrosion resistance, physical properties, and cost. The following discussion is intended to summarize readily available information on corrosion resistance of metals and alloys considered suitable for cooling coil fabrication:

A comprehensive series of tables on composition and properties of corrosion-resistant and heat-resistant metals and alloys were published by the Society in 1924 and 1930 (66). The data given therein furnish an excellent starting point. This has been supplemented by subsequent published data, advices from manufacturers, and experiences of users of various types of coils.

There is no generally resistant metal or alloy unaffected by attack of a corrosive nature. Only the rarer, more expensive metals show resistance to corrosion under a wide range of circumstances, and even gold and platinum are subject to attack under certain conditions. Certain alloys, however, are especially suited for certain imposed situations, and certain of the common metals and alloys may form self-protective films in suitable media thus maintaining effective corrosion resistance (64, 65). It is a dangerous procedure to make an unqualified prediction as to the action of various chemicals on any particular metal or alloy; service tests under actual conditions are the final proof of the performance of the material. It would be impractical and entirely misleading to attempt to tabulate all the metals and alloys as to their ability to withstand the action of all chemicals and concentrations that might be encountered in the waters of sampled steam, boiler water, and cooling water. It is possible, however, to point out the generally resistant properties of certain of the more common metals and alloys and to denote under what environment they have shown satisfactory characteristics. Inquiries regarding applications of newer alloys should be addressed to the technical departments of producers of these alloys, with full and detailed information of the conditions of service and waters so that if suitable information is not available service tests may be made. In any case, the producer of a given metal or alloy should be consulted for details of physical properties of the products under consideration.

The following paragraphs provide a very general discussion of the relation between the sampling of steam and boiler water and the corrosion-resistant properties of iron and steel, including stainless steels; copper, brasses, and bronzes; nickel and monel; aluminum; tin and rare metals.

Iron and Steel:

Iron and steel are commonly used in the sampling of steam and boiler water where pressures and temperatures involved are low, limits of boiler water constituents are wide, and accuracy of tests is nominal. As greater demands are placed on sampling in the higher pressure and temperature range, iron contamination cannot be permitted, and ferrous alloys and non-ferrous alloys and metals are utilized.

Stainless Steels:

Stainless steels find wide application where corrosion resistance is a requisite (11, 17). It is generally agreed that this freedom from corrosion is due to the formation of a protective surface film, a product of reaction between the metal and a corroding medium. Both nickel and chromium, the major alloying elements with iron to produce stainless steels, have the same property of developing surface passivity. Increasing corrosion resistance is obtained in the case of chromium steels by increasing the chromium content.

Stainless steels find successful application in cooling coils for high-pressure boilers in the sampling of steam and boiler waters. Their generally high corrosion resistance to the impurities in boiler water and steam provides a very pure uncontaminated sample for high accuracy measurements. On the cooling water side, some of the stainless steels may show pitting especially where salt or brackish waters with industrial wastes are encountered. Even in these cases the manufacturer may be able to provide a stainless steel modified by the addition of small amounts of silver, molybdenum, copper, or other elements to meet the necessities of the situation. The various producers of this group of alloys have considerable technical information concerning their products, and consultation with their technical representatives is recommended prior to drawing up specifications for stainless steel for the sampling of steam and boiler water for a particular installation. The use of stainless steel for the sampling of steam and boiler water has been reported in the technical literature (29, 46, 58).

Copper and Copper-Base Alloys:

Copper finds a very frequent application in cooling coil construction because of its generally acceptable corrosion resistant properties coupled with its ease of being fashioned into tubular coils (75, 8). Copper owes its corrosion resistance to the formation of a very thin, very tenacious film of cuprous oxide (22). In contact with solutions capable of preserving or reforming this film copper shows remarkable resistance. Consequently it is able to resist oxygen-bearing steam condensate. On the other hand, carbon dioxide and ammonia, two common constituents in steam, can act as active corrosion agents with respect to copper (51). Copper sampling lines and cooling coils have been reported as satisfactory for sampling steam and boiler waters up to a temperature of 406 F. Contamination of steam samples by copper has been observed to continuously decrease to a zero or constant value at the end of a week as shown by measurement of the electrical conductivity of the condensed steam (47). Interruptions in the flow of the condensate were followed by renewal of the contamination until considerable flow had been reestablished. When sea water containing oxygen is used as the cooling medium, copper may be superseded advantageously by the use of red brass, admiralty metal, or aluminum brass as a cooling coil material of construction. On the other hand, these alloys may be subject to dezincification with acid-bearing industrial waters. There are other available copper alloys with distinctive characteristics for special cases.

Nickel and Its Alloys:

Nickel is a relatively noble metal and consequently is but slightly attacked by acids, unless accelerated attack is occasioned by the presence of air or oxidizing agents. On the other hand nickel has the ability to form a passive oxide film under certain kinds of attack. Neutral and alkaline solutions including ammonia are well resisted by nickel so that satisfactory service may be expected with most cooling waters and with neutral or alkaline boiler waters and steam condensate. Cooling water containing acid industrial wastes or acid mine waters should be questioned for use with nickel. Carbonated water may show some attack on nickel so that caution must be used in selecting nickel when steam samples are liable to contain carbon dioxide.

Monel metal, an alloy of copper and nickel, possesses ability to withstand corrosion by many media, being more resistant than nickel under reducing conditions and more resistant than copper under oxidizing conditions. Monel may be used for steam and boiler water generally. Its susceptibility to sulfurous acid makes it advisable to determine its resistance to boiler waters containing sulfite before use in sampling equipment for same. Since monel is resistant to hard and soft natural waters, sea water, and distilled water, trouble would not be expected on the cooling side of the coil except where sea water is allowed to remain stagnant in contact with the monel metal. Monel is not highly resistant to most oxidizing acid salts and where cooling water is contaminated with industrial wastes it may be incompatible.

Aluminum:

The stability of aluminum, a metal which is very active chemically, depends upon the formation of a very thin adherent coating of aluminum oxide. Aluminum has been used to handle steam and distilled water, but it may not be prudent to use this metal with waters having alkalinities much above pH 8.0. A maximum temperature of 400 F. is considered the limit when using aluminum with steam and hot water. Ammonia solutions do not ordinarily attack aluminum unless other salts or alkalis are present. It should not be used where salt or brackish water is used for cooling. Highly alkaline boiler waters could not be handled successfully by aluminum cooling coils. In general, it would be highly desirable to establish the suitability of aluminum by means of service tests under like conditions prior to use. Aluminum alloys are available, formulated to provide definite properties superior to the metal itself. The producers of these alloys are the best source of data on their specific properties.

Tin:

The use of block-tin condensers has long been established for condensing distilled water of high purity. Applications of tin condensers and tin-lined coils in the sampling of steam have been reported (**47**) but the tin linings have been criticized as being uncertain in their protective action against contamination (**70**). Alkaline boiler waters were found to dissolve sufficient tin to adversely affect the colorimetric determination of silica (**58**).

Noble Metals:

The noble metals such as gold, silver, and platinum are relatively corrosion-resistant under most conditions of use. Their cost precludes normal industrial use but recent advances in the technique of applying thin coatings of these metals to cheap common metals expand their availability. It is understood that platinum-clad steel is available for temperatures as high as 1470 F. No instances of this or similar products applied to cooling coil construction are known to the authors.

Metal Combinations Used in Cooling Coil Construction:

Where cooling coil construction provides for an inner tube within which the sample is cooled and an outer tube or shell to provide flow for the cooling water, it is common practice to use copper for the outer tube while the selection of the inner tube depends upon the nature of the cooling water and the sample. Copper, stainless steel, nickel, monel, and 70–30 cupro-nickel, are commercially available in prefabricated cooling coils. Fittings for joining the coils to other piping are selected to avoid metal-couple corrosion. Metals used for fittings in the above cases have been bronze, brass, carbon steel, monel metal, stainless steel, or cupro-nickel for the corresponding service. Naval brass fittings are considered suitable for use with 70–30 cupro-nickel.

Degasifiers:

The use of degasifiers following or integral with cooling coils is often essential in sampling and testing steam condensate; these are not discussed herein as they are outside the scope of this paper (**47, 54, 48, 63**).

VOLUME OF SAMPLE REQUIRED

The volume of sample collected must exceed the sum of the volumes required for the total number of tests to be made thereon. The individual volumes may vary, especially in the case of high-pressure boilers where the concentrations are kept at low levels. Estimates of the volume required for each of various constituents found in industrial waters are given in the A.S.T.M. Tentative Methods of Sampling Plant or Confined Waters for Industrial Uses (D 510 – 40T).[3] These are considered as merely a guide and are subject to modification consistent with directions under methods of analysis.

Apparatus has been developed for the continuous recording of such properties of the boiler water or condensed steam as pH, electrical conductivity, and dissolved oxygen content. Here the sampling equipment must provide a continuous sample sufficiently cooled and especially free from contamination, because of the high degree of sensitivity of these methods. In general a larger volume of sample will be handled in order to reduce the time lag occasioned by travel of the sample through the sampling lines and cooling coils to the measuring and recording apparatus. If the measuring apparatus is designed to handle a small volume of sample the smaller quantity is drawn off from the main sample line by means of a tee at a point near the measuring apparatus. Short sampling lines and minimum cooling capacity with location of the measuring equipment near the point of sampling are further expedients to reduce the inherent time lag.

SAMPLE CONTAINERS

The sample container must preserve the quality of the cooled sample, sometimes over an extended period of time; hence its construction must prevent outside contamination and at the same time avoid adding undesirable constituents by solution from within the sampling equipment. The requirements for sample containers are less stringent in respect to high temperature reactions and the container is not exposed to the attack of the cooling water. On the other hand the sample container may be exposed to possible attack by slow reaction of the cooled boiler water or condensed steam over a long time, resulting in the formation of corrosion products which may be sufficiently soluble to appreciably contaminate the sample, or reducing the concentration in the sample of the active corrosion agent.

Choice of material for the sample container will be governed by the nature of the water or steam being sampled, the nature and extent of the contamination by the material, and finally whether such contamination can be (1) removed subsequently, (2) quantitatively determined and correction applied, or (3) completely disregarded if proved to have no effect on the property being measured.

Materials for Sample Containers:

Consideration of the metals discussed for cooling coil construction indicates that the same materials may be used for sample containers under similar condition of service, with suitable attention paid to the foregoing factors pertaining specifically to the containers.

Ordinary glass may be used as a material of construction for the sample container in many cases, such as routine boiler water samples, where slight attack of the sample water on the glass over a short time is not harmful. Committee D-19 on Water for Industrial Uses has recommended for sampling of industrial waters[4] the use of clean, narrow-mouth glass bottles; preferably glass-stoppered, although new corks may be used. It is noted that highly alkaline waters may dissolve silica from certain types of glass containers. One method of counteracting this solubility, where silica is being determined, is to make the sample definitely acid with a known quantity of sulfuric or other acid and later to apply the necessary corrections. For waters or condensed steam having a pH of 6.0 or over, the bottle glass should be of good chemically resistant type. Ordinary glass bottles, but not the cork, may be coated internally with paraffin to protect the sample from the glass.

The use of tin and tin-lined containers for the collection of samples of boiler water and condensed steam has been reported (70, 56). Ordinary 5-gal. milk cans were found to be unsatisfactory unless recoated with a heavier tin layer and the seams well sealed. Samples of condensed steam have been collected in a 500-ml. silver flask closed with a silver stopper but appreciable amounts of silver were found in the sample (58). The use of platinum will generally be precluded by its cost but the development of platinum-clad metals may offer the requisite strength at a lower cost. Hard rubber bottles after a short period of aging have been suggested as being a suitable container for the transportation of alkaline boiler waters to avoid the use of tin or glass (56).

Besides the use of pure metals as a protective coating, lacquers, varnish, paraffin, and other applied coatings may offer promise for protection of the base metal from corrosion and the sample from contamination. It is imperative that the coating be continuous, without pinholes, and of sufficient thickness to give suitable life to the coating. Furthermore, the chemical stability of the coating must be established for the particular use. Naturally forming coatings which are self-healing in contact with the water are the best form of protection.

CONCLUSIONS

The sampling of steam and boiler water involves common-sense application of basic sampling principles to specific local problems. Choice of sampling locations, methods of sample withdrawal, and volume of sample can be selected from a clear understanding of the reasons for sampling, tests to be made, accuracy necessary, and similar considerations. Choice of cooling equipment becomes largely a consideration of materials of construction from the standpoints of strength, corrosion resistance, and degree of sample contamination allowable. Available data on metals and alloys is not particularly specific as regards their usefulness with steam, boiler water, and the various types of cooling water available.

Limited available data, plus general information on the properties of ferrous and non-ferrous materials, indicates that stainless steel, copper and copper alloys, and nickel and its alloys have definite fields of usefulness in the sampling field, but a clear knowledge of the chemical constituents present in sample and cooling water is essential to proper choice of material, and discussion of the potential use with the metal producer is considered an advantageous procedure to insure correct choice. It is suggested that users of cooling coils augment the present limited data by publishing their experiences in detail and in a uniform manner. Such detail should include the following: pressure, temperature, and chemical composition of the steam or water sampled; temperature, relative rate of flow, and chemical analysis of cooling water; complete chemical and physical properties of metals or materials of construction; details of design of coils and their cooling capacity; type of service required of the sampling equipment.

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[4] See Tentative Methods of Sampling Plant or Confined Waters for Industrial Uses (D 510 - 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 523.

Sampling containers may be selected in similar manner with a full understanding of requirements. The obtaining of data on the usefulness of the newer lacquer type coatings should be considered, as these may offer an economical means of preparing sample containers. Other data which would aid in proper selection of coils and containers are the limiting maximum permissible concentrations of interfering substances in each method of test.

DISCUSSION

Mr. Max Hecht[1] (*by letter*).—Industry in general, and Committee D-19 on Water for Industrial Uses, in particular, will evaluate the contribution of the authors, for its factual completeness and thoroughness of the analysis of the problem assigned to them. The writer confines himself to the phase of the paper that deals with the apparatus required for sampling, particularly the sampling of the fluid within the pressure vessel and pipe lines.

A rule followed in the sampling of finely divided materials in suspension is to sample the whole of the stream, part of the time; when the various portions are accumulated and mixed this represents a true sample of the entire bulk sampled. This rule cannot be followed in the sampling of steam or water within a pressure vessel, particularly at elevated pressures and temperatures. The large size of the modern steam generator, and the cross-section of the steam offtakes do not lend themselves readily to the insertion of a sampling tube to secure a representative sample. Thus, to approximate the rule above stated, a truly representative sample may be secured by sampling the generator at multiple points along the length of the vessel or sampling the steam in each offtake where multiple offtakes are provided. Hecht **(25)**[2] shows that concentration of boiler water in the drum varies along the length of the boiler drum, and concludes that circulation of water is largely confined to vertical paths. Baker **(3)** shows that the steam purity varies across the boiler, and that the poorest quality is found in the offtakes located at the center of the boiler. Taking these two independent observations together, it may be noted that steam, as generated, is a heterogeneous mixture of gas and water droplets. Furthermore it has been stated by Cassidy **(5)** "The actual maximum steam velocity in modern high-capacity boiler drums may generally exceed the critical, so that any moisture picked up from the surface of the water will be carried by the steam except at reduced outputs." Cassidy shows the variation in the critical velocity of steam with pressure (Stokes' law) in his Fig. 24. The formula used by Cassidy is discussed rather completely by Hall **(23)**. Place[3] states: ". . . the amount of gases may vary considerably because the gas carry-over from the boiler is not a function of the moisture carry-over but rather a function of the rate of gas liberation in the boiler." Thus it may be concluded that boiler water should be sampled in the drum from which the steam is liberated, and as close to the normal operating water level as is practical, and furthermore that the steam sample should be collected from multiple points within the steam drum as close to the steam offtakes as is practical.

The authors point out that a sample-gathering pipe having openings of equal area will select a sample adjacent to the opening nearest to the container wall. (It should be noted that the nozzle specified in the A.S.M.E.Test Code (13) has openings of uniform area, and has been used for steam sampling by some of the investigators on the general subject of steam quality.) In many cases the free flow of steam through the nozzle has been restricted and adjusted to satisfy the volume requirement for test. If the A.S.M.E. Test Code requirements are fulfilled as to the free flow of steam to the atmosphere, as specified for the calorimeter test, it is probable that a representative sample can be secured for the requirements of the electrical conductivity test. Baker (3) appears to have satisfied these conditions by using a number of nozzles, diverting a part of the steam stream to the atmosphere, and condensing part of the stream for his conductivity measurements. Other investigators on this subject (mentioned in the bibliography to the papers included in this symposium[2]), omit detailed information as to steam velocities in the steam mains sampled, and steam velocities maintained through the sampling nozzle and cooling equipment.

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[1] Adviser, Power Stations Chemistry, Pittsburgh, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

[3] P. B. Place, "The Degasification of Steam Samples for Conductivity Tests," see p. 1302.

Tubing of small internal area has been suggested in current practice for condensing and cooling coils. Caution should be observed, in specifying the size of tubing, to avoid linear velocities exceeding the critical, so that extensive dissolution of the metal will not occur. If excessive velocities above the critical are maintained and the oxygen content is low, the protective film on some of the corrosion-resistant alloys may be eroded, thus contaminating the sample.[4]

In answer to the authors' plea for operating evidence on sampling technique, the writer supplies the following:

The results of a traverse made on a 250-psi. cross drum horizontal tubular boiler showed rather conclusively that the concentration of dissolved gases as well as dissolved salts in the water cycle varied across the boiler. Sample tubes were fitted into the boiler caps on each fifth tube on a boiler 52 tubes wide. Details of this test are given by the writer (25). The steel sample tube in the drum, extended across its length and submerged below the water line, was perforated with openings of unequal area, the smallest opening being nearest to the cooling coil inlet. The sizes of the openings were calculated to secure equal volumes of water along the length of the drum. All facilities for condensing steam and cooling of condensate and boiler water were of copper, and the rate of flow was such as predicated for securing the oxygen sample, namely one liter per minute.

In the conductivity investigation by Hecht and McKinney (27), the steel boiler water sample line within the drum was similar in design to that referred to above, and the steam sample nozzle (A.S.M.E. standard type) was inserted in a large saturated steam lead between the drum and the superheater header. Free flow through the nozzle was maintained. In this test installation, the apparatus within the boiler and steam main were steel, and the cooling coils were copper. All leads to the coils were very short. The flow rate of cooled boiler water and steam condensate was controlled at 1 liter per minute. Houghton (27), reporting to the discussion to this paper, appears to confirm the experiences of the cited investigation, that concentrations of salts varied in the boiler water across the drum. In the closure of the investigation (27), the authors include observations made at another plant wherein an improved design of steam sampling nozzle was provided.[5]

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[4] For a discussion of the relation of velocity of slow and corrosion rate for various temperatures and pipe sizes, the reader is referred to Speller and Kendall (61), also to Speller (60).

The accompanying Fig. 1 is a photograph of the nozzle inserted in a 24-in. steam lead from the steam offtake drum. This nozzle was of corrosion-resistant alloy. The sample line for the boiler water was of steel, perforated with openings of unequal area, the smallest area being adjacent to the cooling coil inlet. Copper cooling coils were provided, the one for steam was located immediately at the steam lead sampling point, the one for boiler water some 30 ft. away.

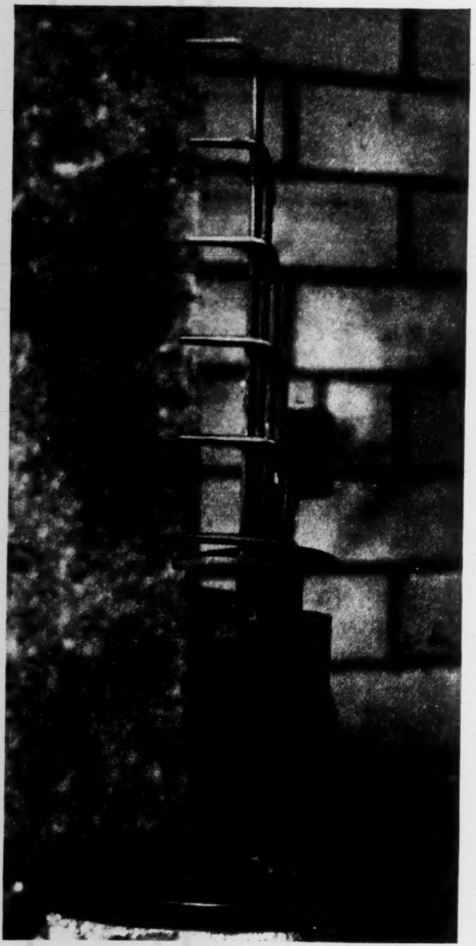

FIG. 1.—Campbell Steam Lead Sampler.

Subsequent to the above reported tests, installations of similarly designed steam samplers were made at another operating station. The boilers, operating at 440-psi. pressure, were of multidrum design, with bent tubes. In this test installation the multipoint sampler for steam was slightly different in design, that is, the length of the tubes was increased, and the assembly was installed adjacent to and parallel to the steam offtake pipe within the drum. Similarly designed sampling devices, having tubes of differing lengths were installed in several of the drums, the object being to secure a representative sample from along the length of each drum for both steam and boiler water. The material used for the sampling devices within the drums was of corrosion-resistant alloy. The drum walls required but one opening to accommodate this device. Cooling coils of copper were located immediately adjacent to the respective drums. This installation was provided with a back washing line using the feedwater pressure for cleaning the sampling tubes of any boiler water deposit accumulations.[6]

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[5] Eugene G. Campbell, "Apparatus for Treating and Sampling of Substances," U. S. Patent No. 2,030,682, issued February 11, 1936.

[6] The late E. G. Campbell, Betterment Engineer, Power Stations Dept., Duquesne Light Co., Pittsburgh, Pa., designed the multiorifice sampler to meet the more exacting requirements of securing the steam sample for the electrical conductivity test. The principles governing the design are fully explained in the specification to the patent, and the drawings included show various designs for the collection of steam and water samples in equal proportions along the length of the drum, as well as across the area of a steam lead, and requiring but one opening in the wall of the drum or steam lead for introducing the flowing water or steam sample to the cooling and condensing coils.

EXPERIMENTAL METHODS OF DETERMINING CONDUCTIVITY CORRECTIONS FOR DISSOLVED GASES IN STEAM CONDENSATE

By S. F. Whirl[1] and W. A. Lower[1]

Synopsis

This paper presents a critical survey of the various methods that have been advocated for correcting conductivity measurements on steam condensate samples for the presence of interfering dissolved gases, principally ammonia and carbon dioxide. In general, the basis of these methods has been either the application of correction factors for the amounts of ammonia and carbon dioxide present, as determined by analysis, or, an attempt at gas removal prior to the conductivity measurement. Because of inaccuracies or involved analytical procedures, none of them is entirely satisfactory.

To avoid the inherent errors of these methods, a fundamentally different procedure has been developed in the authors' laboratory. The gases are distilled from the sample, which is made successively alkaline and acid, into relatively pure water and the conductivity measured. Subtraction of a blank determination value then gives directly a single correction for the combined effects of all gases. No analyses for conductive gases are required.

Of the various procedures that have been employed for determining steam purity, the electrical conductivity method, because of its extreme sensitivity and its adaptability to continuous measurements, is generally recognized to be the best and is, therefore, the one in most general use. Outstanding in the development of the method was the pioneer work of Fitze (**18**),[2] Hecht and McKinney (**27**), and Rummel (**53**).

The chief disadvantage of the method is the error resulting from the presence of dissolved gases such as ammonia, carbon dioxide, hydrogen sulfide, and sulfur dioxide. Gases, other than ammonia and carbon dioxide, have received little study, however, since they are usually present only in negligible quantities. Since these gases form ions in solution, the apparent concentration of dissolved salts may be several fold too high if they are disregarded. In adapting the electrical conductivity method to actual practice, some means of correcting for such gases has been found imperative. Particularly is this true when the dissolved salts content of the steam is less than 1 ppm.

A number of different procedures for eliminating the effects of the conductive gases has been devised. In general, two lines of approach have been followed: first, the application of predetermined conductivity correction factors based on analytical test data and, second, physical removal, as far as possible, of the gases prior to the conductivity measurement. Both methods have been hampered by certain inherent difficulties, namely, in the first, uncertainty as to the proper factor to be used for ammonia and carbon dioxide and, in the second, lack of a practical means of completely removing the gases.

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[1] Chief Chemist, and Chemical Laboratory Supervisor, respectively, Duquesne Light Co., Pittsburgh, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

TABLE I.—EFFECT OF EVAPORATION ON ELIMINATION OF AMMONIA FROM AQUEOUS SOLUTIONS.[a]

Solution	Volume of Sample, ml.	Ammonia, as NH_3, ppm.[b]	Volume After Evaporation, ml.	Ammonia, as NH_3, After Evaporation, ppm.[b]	Ammonia, NH_3, Retained in Solution, per cent
$(NH_4)_2SO_4$	500	0.1	248	0.14	68.0
	500	0.1	236	0.14	66.0
	500	0.5	234	0.64	60.0
	500	0.5	240	0.64	61.6
	500	1.0	240	1.50	72.2
	500	1.0	236	1.44	67.8
	500	0.1	119	0.35	84.0
	500	0.1	109	0.37	80.0
	500	0.5	125	1.78	89.2
	500	0.5	107	2.11	90.4
NH_4Cl	500	0.1	232	0.16	76.0
	500	0.1	235	0.16	76.0
	500	0.5	254	0.59	59.6
	500	0.5	230	0.60	55.6
	500	1.0	232	1.66	77.2
	500	1.0	260	1.47	77.4
	500	0.1	94	0.41	78.0
	500	0.1	100	0.39	78.0
	500	0.5	116	1.84	85.6
	500	0.5	109	2.11	92.0
$NH_3 - CO_2$	500	0.1	246	0.12	60.0
	500	0.1	245	0.12	60.0
	500	0.5	248	0.26	26.0
	500	0.5	256	0.26	26.8
	500	1.0	240	0.23	11.2
	500	1.0	255	0.24	12.4
	500	0.1	112	0.05	12.0
	500	0.1	110	0.04	8.0
	500	0.5	122	0.04	2.0
	500	0.5	108	0.06	2.4
NH_4OH	500	0.1	235	0.09	39.2
	500	0.1	223	0.05	24.0
	500	0.5	215	0.06	4.8
	500	0.5	225	0.08	6.8
	500	1.0	225	0.11	5.0
	500	1.0	240	0.12	5.8
	500	0.1	118	0.03	8.0
	500	0.1	112	0.04	8.0
	500	0.5	119	0.05	2.4
	500	0.5	105	0.06	2.4

[a] Laboratory prepared solutions.

[b] By direct Nesslerization with clarification procedure omitted. Standard Methods of Water Analysis, Am. Public Health Assn., Eighth Edition, p. 44 (1936).

Hecht and McKinney (27) early recognized the necessity for correcting conductivity values and developed a carbon dioxide correction curve by the preparation and testing of synthetic solutions. At that time these investigators did not correct their results for other interfering gases, but mentioned that "it should be noted that hydrogen sulfide (H_2S) and ammonia (NH_3) also increase the conductance, and, if present, corrections must be applied." Later (26), due to the known presence of ammonia, correction was made for all gases by evaporating the sample to one half its original volume and cooling to room temperature before making the conductivity measurement. This procedure, of course, altered the factor used in correlating the conductivity readings and dissolved salts content. Baker (3) in correcting for ammonia reduced the sample to one fifth its original volume before determining the conductivity.

It has been the authors' experience that serious errors may be encountered when this "boiling-down" technique is employed, due to failure to remove a considerable portion of the ammonia. Some results obtained with various prepared solutions are given in Table I. It is to be particularly noted that in many cases the ammonia is actually concentrated somewhat, hence, the conductivity due to dissolved gases would be greater than in the original sample.

A more refined evaporation procedure was later developed by Place (45) in which he recommended distilling a given volume of sample in a glass still until the distillate indicated low carbon dioxide content and minimum conductivity. The portion of the sample remaining in the flask was then assumed to be gas free. By determining its volume and conductivity, the dissolved salts content of the original sample was calculated. It is obvious that this method is subject to the same errors involved in the "boiling-down" procedure described above.

Various distillation methods for degasifying a continuously flowing sample have been advanced by Rummel (**53**), Powell (**46, 47**), Gurney (**20**), Joos (**29**), and Straub (**63**). In general, as pointed out by Schwartz and his associates (**57**), equipment based on this principle will not completely remove ammonia. Straub, however, has recently reported reductions in ammonia from 3.5 to 0.01 ppm. with his equipment.

The principle of aeration has also been used to effect gas removal from the condensate sample. Powell (**47**) and Betz (**4**), using this treatment, developed equipment for producing a continuous sample. Powell, however, has expressed a preference for his vacuum-distillation procedure. Smith and Williams (**59**) in their laboratory method recommended passing purified air through the sample for 30 min. before making the conductivity measurement. The sample was then tested for residual ammonia and a correction factor applied.

TABLE II.—CONDUCTIVITY OF IONS.

Ion	Milliequivalents per Million	Ionic Equivalent Conductivity, mhos per equivalent at 25 C.	Conductivity per ppm., mhos per cm. at 25 C.
Hydrogen, H^+	0.9921	349.8×10^{-6} [a]	347.04×10^{-6}
Hydroxide, OH^-	0.0588	198.0[a]	11.64
Ammonium, NH_4^+	0.0587	73.4[a]	4.31
Bicarbonate, HCO_3^-	0.0164	44.5[a]	0.73
Carbonate, CO_3^{--}	0.0333	83.0[b]	2.76

[a] D. A. MacInnis, "The Principles of Electrochemistry," p. 342, Reinhold Publishing Corp., New York, N. Y. (1939).

[b] F. H. MacDougall, "Physical Chemistry," p. 477, The Macmillan Co., New York, N. Y. (1936).

In the application of separate conductivity correction factors for ammonia and carbon dioxide in accordance with an analysis of the sample for these gases, negative values for dissolved salts are often obtained. In the authors' laboratory this anomaly has been found to occur particularly on samples which contain relatively high concentrations of both gases. The reason for such erroneous results is the failure to consider the influence of varying amounts of ammonia and carbon dioxide on the relative concentrations of H^+, OH^-, NH_4^+, HCO_3^-, and CO_3^{--}. Of particular significance is the effect of the H^+ and OH^- concentrations, since the conductivity per ppm. of these ions so greatly exceeds that of the others, as shown in Table II. Therefore, the true conductivity correction for ammonia and carbon dioxide should be a single factor which takes into account the equilibrium relationship resulting from the effects of these gases on each other and on the sample.

Lack of consideration of this relationship is believed to be responsible for the disagreement among investigators as to the proper correction factor to use for ammonia. When calculated as conductivity in micromhos per part per million of ammonium ion, NH_4^+, the following values have been reported: Rummel,[3] 5.6; Powell (**46**), 7.0; Schwartz (**57**), 6.2; Smith (**59**), 5.7; and Ulmer (**70**), 7.0.

Recognizing the significance of the ionic equilibrium existing at the time the conductivity of the sample is determined, Watson (**74**) has proposed an interesting and logical method whereby a single correction is determined from a family of curves relating the pH value and various concentrations of the gases. The curves were formulated from the dissociation constants of ammonium hydroxide and carbonic acid. His suggested procedure is as follows:

(*a*) The concentration of ammonia and "free" carbon dioxide are determined.

(*b*) If the "free" carbon dioxide is zero or if the pH value exceeds 7.0, the pH is adjusted to some value below 7.0

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[3] From curve prepared by J. K. Rummel and distributed through the courtesy of the Babcock & Wilcox Co.

by addition of carbon dioxide, after which the ammonia content is redetermined.

(*c*) The conductivity of the sample under these conditions is then measured and a correction factor, taken from the curves relating pH value to gas concentration, is subtracted from this observed value, the corrected conductivity then being converted into parts per million of dissolved salts. As shown by Watson, this method appears to eliminate the negative values for the dissolved salts content which are often obtained when using separate correction factors for the two gases.

In any method based on the results of analytical tests for ammonia and carbon dioxide in the sample, the importance of accuracy cannot be overemphasized. Due to the high conductivity corrections for these gases, seemingly small errors in the analyses may produce major discrepancies in the results, especially when the dissolved salts concentrations are in the order of 0.1 to 0.5 ppm., as have been reported by some investigators (**3, 70, 58**). This is particularly true for ammonia, since relatively small changes in the concentration of this gas considerably alter the conductivity of the sample.

The Nessler method,[4] either by direct or distillation procedure, is used almost universally for the determination of ammonia. A study of this method in the authors' laboratory has shown that freshly prepared standards are distinctly superior to the permanent ones; they compensate for variable sensitivity of the reagent. It has also been noted in the determination of small quantities of ammonia by the distillation procedure that the accuracy of the results is improved by protection of the sample and distillate from atmospheric contamination and by matching colors in equipment that provides standard light conditions. The work of Schwartz and his associates (**57**) includes a thorough investigation of the Nessler and other methods. Their data relative to the volumetric method proposed by Teorell (**68**) using sodium hypobromite with naphthyl red as an indicator is of particular interest.

For the determination of "free" carbon dioxide, the method which is generally used is some modification of the A.P.H.A. procedure.[5] The size of the sample, the standard alkali reagent, and the strength of the standard solutions have varied with different investigators. For the concentration usually encountered in steam, it is believed that this method is not satisfactory, the results being especially questionable for values below 1 ppm. The total carbon dioxide is best determined by the evolution method of Partridge and Schroeder (**42**),[6] or some modification of this procedure. The turbidimetric adaption proposed by Roller and Ervin (**52**) appears to be a worthwhile contribution.

A new method of correcting conductivity for the influence of dissolved gases has been developed in the authors' laboratory. No chemical tests are required and a single correction for the combined effects of all gases in the sample is obtained. In this method the gases are transferred by distillation into relatively pure water in which their influence on conductivity is determined. To assure complete removal of the gases, the sample is first treated with an alkali and then with an acid during the distillation procedure. A diagrammatic sketch of the apparatus is shown in Fig. 1.

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[4] Standard Methods of Water Analysis, Eighth Edition, pp. 41-45, Am. Public Health Assn. (1936).

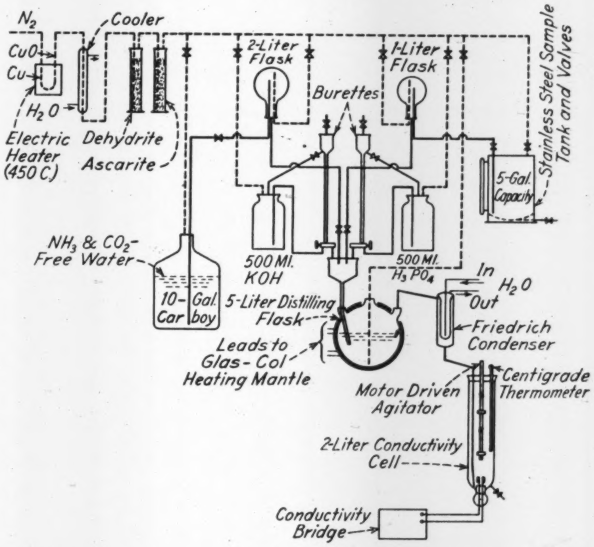
[5] Standard Methods of Water Analysis, Eighth Edition, p. 69, Am. Public Health Assn. (1936).

[6] Tentative Method for Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (D 513 – 38 T), 1939 Book of A.S.T.M. Standards, Part III, p. 1108.

DESCRIPTION OF APPARATUS

Distillation Assembly:

The distillation flask is a three-neck, round-bottom, pyrex vessel having a capacity of 5000 ml. The sample and chemical treatment are admitted through a feed line which extends well below the minimum water level experienced during distillation. An aeration tube, the end of which is near the flask bottom, is provided for purging the flask, Friedrichs condenser, and conductivity cell with purified nitrogen. All connections to the flask and condenser, including the condensate delivery tube, are made with interchangeable ground-glass joints.



N_2
Cooler
CuO
Cu
H_2O
Electric Heater (450 C)
Dehydrite
Ascarite
2-Liter Flask
Burettes
1-Liter Flask
Stainless Steel Sample Tank and Valves
5-Gal. Capacity
NH_3 & CO_2-Free Water
10-Gal. Carboy
500 Ml. KOH
500 Ml. H_3PO_4
5-Liter Distilling Flask
In
H_2O
Out
Friedrich Condenser
Leads to Glas-Col Heating Mantle
Centigrade Thermometer
Motor Driven Agitator
2-Liter Conductivity Cell
Conductivity Bridge

FIG. 1.—Apparatus for Determining Conductivity Correction for Dissolved Gases in Steam Condensate.

The flask is heated by means of a Glas-Col heating mantle which completely envelopes the vessel. This provides uniform heating and also prevents condensation and reabsorption. The vapor offtake, which extends vertically a distance of 9 in. above the flask opening, is lagged with asbestos tape up to the point at which the vapor enters the condenser.

Conductivity Cell:

The conductivity cell is a round-bottom, cylindrical pyrex chamber of 2000-ml. capacity. Platinized platinum electrodes, adjusted to a 0.1 cell constant, are mounted in a ground-glass plug which is inserted in the bottom. The condensed vapors are received through a tube which extends to the bottom of the cell. Thorough mixing of the accumulated condensate is accomplished by means of a motor-driven agitator.

Conductivity Bridge:

The bridge used in measuring conductivity is a Leeds & Northrup, 110-v., 60-cycle instrument, Catalog No. 4866, which operates over the range 0.1 to 12,000 micromhos. Variations in cell constant and temperature are corrected by manually operated compensators.

Nitrogen Purification:

As indicated by the broken lines in the sketch, the entire system is provided with complete protection from atmospheric contamination by a purified nitrogen atmosphere. Nitrogen is also used in transferring the sample and reagents. Purification of the nitrogen is accomplished by passage through a tube containing copper and copper oxide heated to a temperature of 450 C. and subsequently through Dehydrite and Ascarite.

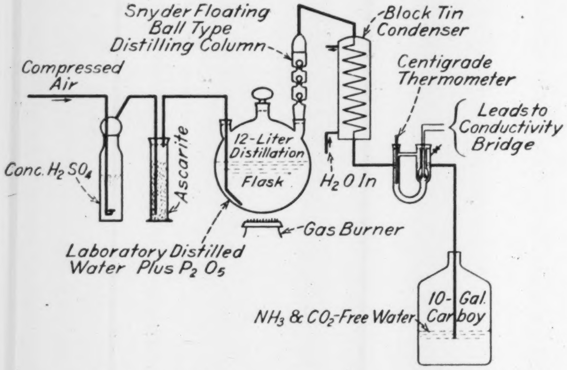

Snyder Floating Ball Type Distilling Column
Block Tin Condenser
Compressed Air
Centigrade Thermometer
Leads to Conductivity Bridge
Conc. H_2SO_4
Ascarite
12-Liter Distillation Flask
H_2O In
Gas Burner
Laboratory Distilled Water Plus P_2O_5
10-Gal. Carboy
NH_3 & CO_2-Free Water

FIG. 2.—Apparatus for Obtaining Water Free of Ammonia and Carbon Dioxide.

Sample Container:

In fabricating the container in which the samples are taken, stainless steel was employed because of its anti-corrosion characteristics. A 5-gal. container was built with the expectation that the data obtained by this method would be correlated with referee gravimetric tests. It is to be particularly noted that in the distillation tests the sample is transferred to the flask directly from the container in which it was taken in the field.

PROCEDURE

Prior to each test, the distillation assembly and conductivity cell are thoroughly purged with purified nitrogen. The distilling flask is then charged with 2 liters of water free of ammonia and carbon dioxide and 1 liter of the sample by means of pressure from the purified nitrogen system. The distillation is started and, as soon as the condenser is thoroughly wet, 4 ml. of 0.1 *N* potassium hydroxide[7] are admitted, giving a pH value of approximately 9. When 500 ml. have been distilled into the conductivity cell, 6 ml. of 0.1 *N* phosphoric acid (27) are added to the flask, thereby lowering the pH to about 4. The distillation is then continued until the volume in the cell is 1000 ml., at which time the conductivity is measured. By subtracting from this reading the value of a blank determination on the reagents and water used, the conductivity correction for the gases in the sample is obtained.

PREPARATION OF WATER FREE OF AMMONIA AND CARBON DIOXIDE

The water free of ammonia and carbon dioxide used in this investigation was prepared by redistillation of laboratory distilled water treated with phosphorus pentoxide. A sketch of the apparatus is shown in Fig. 2, which is self explanatory. It varies from the procedures of Ellis and Keihl (15) and Cranston and Brown (9) in that purified air is bubbled through the liquor instead of the distillate. After trying several other methods, this one was adopted because of the consistently low conductivity values that were obtained. Values higher than 0.2 micromho at 25 C. were seldom experienced, and often this figure was less than 0.1 micromho, the minimum amount which could be measured on our bridge. It was interesting to note that cutting off the purified air supply caused the conductivity rapidly to increase several fold and then diminish at a similar rate when bubbling was started again. While water of this quality is not essential for plant work, it serves to increase the accuracy of the results by giving low values for the blank determinations.

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[7] Prepared by dissolving a "reagent grade" chemical in water free of ammonia and carbon dioxide, boiling the solution, and subsequently cooling under a protected atmosphere.

DISCUSSION AND CONCLUSIONS

The results of several tests with samples containing varying amounts of ammonia and carbon dioxide are shown in Table III. In those tests where "free" carbon dioxide is reported, its determination was effected by titration of a 100-ml. sample in a closed container with $N/132$ sodium hydroxide, using phenolphthalein as an indicator. This procedure was employed because it is the only relatively simple method which gives "free" carbon dioxide, the value used by most investigators. The more accurate methods measure total carbon dioxide. For tests Nos. 1, 2, 8, 9, 10, 13, and 16, synthetic samples were prepared by the addition of measured amounts of standard ammonium sulfate and sodium carbonate solutions. Tests using these samples were made to ascertain the effect of total carbon dioxide on conductivity, since the corrections obtained by the proposed method are influenced by both the combined and "free" gas.

In reviewing these data it is to be noted that the calculated conductivity corrections based on separate factors for ammonia and carbon dioxide are higher, in all cases, than those obtained experimentally. This method, therefore, will tend to eliminate the negative values for dissolved salts, which, as pointed out above, are often encountered.

TABLE III.—CONDUCTIVITY CORRECTIONS FOR DISSOLVED GASES.

Test	Ammonia (NH_4^+), ppm.	Carbon Dioxide (CO_2), ppm.		Conductivity Correction, micromhos at 25 C.	
		Total[a]	Free[b]	Obtained	Calculated[c]
No. 1	0.30	0.5	...	1.54	...
No. 2	0.31	1.0	...	1.71	...
No. 3	0.33	...	0.5	1.91	2.66
No. 4	0.33	...	1.0	2.07	2.91
No. 5	0.41	...	2.0	2.88	3.97
No. 6	0.41	...	2.0	3.11	3.97
No. 7	0.41	...	4.0	3.53	4.67
No. 8	0.61	0.5	...	3.55	...
No. 9	0.61	1.0	...	3.74	...
No. 10	0.61	2.0	...	3.92	...
No. 11	0.61	...	2.0	4.17	5.37
No. 12	0.61	...	4.0	4.57	6.07
No. 13	0.83	1.0	...	5.05	...
No. 14	0.83	...	0.5	5.10	6.16
No. 15	0.83	...	1.0	5.22	6.41
No. 16	0.83	2.0	...	5.40	...
No. 17	0.83	...	2.0	5.61	6.91
No. 18	0.83	...	4.0	5.90	7.61

[a] Determined by the preparation of synthetic samples to which were added measured amounts of standard sodium carbonate solution.

[b] Determined by titrating 100-ml. samples with $\frac{1}{132}$ N sodium hydroxide, using phenolphthalein as an indicator.

[c] Calculations were made by using a factor of 7.0 micromhos per ppm. of ammonium ion and Rummel's curve (53) for carbon dioxide.

In the proposed procedure, the volume of the distillate, in which the conductivity due to dissolved gases is measured, is always the same as that of the sample. This practice was established because the conductivity due to the gases is not directly proportional to their concentration. Accordingly, widely divergent results are obtained if volume correction factors are employed. It is likewise not permissible to use a constant conductivity value for varying concentrations of the interfering gases. This condition is also shown quite clearly in the data presented by Watson (**74**).

While the precautions as noted, namely, the long vertical offtake tube and the method of heating, have been taken to prevent carry-over from the distilling flask, a small amount undoubtedly does occur. It is reasonable to assume, however, that this value is constant and, therefore, included in the blank determination. The check results which have been obtained tend to substantiate this assumption. The use of the blank determination value also compensates the final results for any gases present in the reagents and water used and the conductivity of water itself.

It is recognized that the results of these tests are not in complete agreement with those reported by other investigators. They are not intended as proof of the method, but merely as an indication of its performance as compared with that of those procedures involving analytical testing. It must be borne in mind that the principle feature of the proposed method is the elimination of all chemical tests. To determine the accuracy of the method, it is apparent that its results must be correlated with precise gravimetric data. Such a program of study is contemplated for the near future. It was for this reason that the 5-gal. stainless steel sample container, shown in Fig. 1, was obtained.

For the gravimetric study, an apparatus similar to that of Call's[8] is being constructed. Elaborating on the principles employed by Powell (**46, 47**), Ulmer (**70**), and Schwartz (**58**), Call has developed a method, the essential features of which are automatic control, protected atmosphere, radiant heat, and admission of the cool water at the bottom of the platinum dish through a funnel-shaped, side-arm delivery tube. Its particular advantage is a faster rate of evaporation without disturbance of the water surface.

In adapting the proposed method to general plant use, it is apparent that the apparatus shown in Fig. 1 can be considerably simplified. Sacrificing some degree of accuracy, it is possible to eliminate partially or completely the system for protecting against atmospheric contamination. Likewise, the size of the apparatus may be reduced and the method of adding the sample and reagents altered.

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[8] R. G. Call, private communication.

[See Joint Discussion, p. 1296.—ED.]

CALCULATION OF CORRECTIONS TO CONDUCTIVITY MEASUREMENTS FOR DISSOLVED GASES

BY D. S. MCKINNEY[1]

SYNOPSIS

Methods for calculating the corrections to be applied to conductivity measurements are presented, based partly upon the author's earlier paper "The Calculation of Equilibria in Dilute Solutions" (**37**). See also (**1, 38**).

A procedure is outlined for correcting the conductivity of condensed steam when analysis of steam only is available.

An improvement of the method developed by A. Watson is proposed.

A method for calculating the correction to be applied to a steam sample from partial analysis of steam and boiler water is described and illustrated by an example.

Tables of data needed for the calculations are presented showing (1) the ionic equivalent conductance λ_0 of the ions commonly found in industrial water; (2) the variation of the equivalent conductance Λ with concentration for Na_2SO_4, NaCl, and NaOH; and (3) the distribution of the ions of the weak electrolytes H_2O, H_2S, CO_2, SO_2, NH_3, and H_3PO_4 as a function of pH.

Conductivity measurements for the detection of contamination in relatively pure water, such as steam and condensate returns, have been widely used because of the speed of the method relative to evaporative methods. The necessity for correcting the measured conductivity for dissolved gases such as carbon dioxide was pointed out by Hecht and McKinney (**27**),[2] who described a method for making the correction when only one gas was present. Although this empirical method of correction could be extended to two or more gases, a large amount of experimental work would be required to establish the necessary correction curves. Other workers, for example Rummel (**53**), Powell, Bacon, McChesney, and Henry (**47**), Gurney, Schwartz, and Crossan (**20**), and Straub and Nelson (**63**), have developed apparatus for removing a substantial portion of the offending substances by distillation. While these devices are of great value, since they simplify the problem of interpreting the conductivity data, there will be many situations where they are not necessary if the corrections can be made in other ways.

The problem of making sufficiently accurate corrections for the conductivity due to dissolved gases has been made more difficult in recent years by the improvement in the quality of steam, due to more careful water treatment, better operation and design of boilers, and by the increased number of cases in which gases such as NH_3 produce the greater part of the measured conductivity. Ulmer (70) reports contamination due to NH_3 sufficiently great to make the condensed steam alkaline to phenolphthalein. When the contamination is so great, the use of the devices mentioned above, for removal of offending substances by distillation, is suggested to reduce the correction at least to the order of magnitude of the conductivity due to the nonvolatile substances present.

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[1] Assistant Professor of Chemistry, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

Since a complete discussion of all types of contamination would require a very long paper, the subject matter of this paper is limited to the determination of entrainment of boiler water in steam by conductivity methods.

ASSUMPTIONS AS TO ACCURACY OF MEASUREMENTS

In order to interpret the conductivity measurements in terms of either nonvolatile salts in the steam or percentage carry-over from the boiler water, it is necessary to eliminate contamination by the sampling apparatus and to accurately measure or determine the following:

1. The true conductivity. Polarization effects must be eliminated by use of small currents through the cell, use of alternating-current bridges, and proper platinization.
2. The volatile substances, such as NH_3 and CO_2, which affect the conductivity.
3. The nonvolatile weak electrolytes.

In the measurement of conductivity, accurate temperature compensation must be provided. Although the temperature coefficient of conductivity is nearly the same for all ions, it should be remembered that the dissociation constants of weak electrolytes change rapidly with temperature, thus changing the ion concentrations. When the conductivity due to volatile substances is large compared to that due to nonvolatile salts, measurements should be made at 25 C. or as near this temperature as possible.

RELATION OF CONDUCTIVITY CORRECTIONS TO EQUILIBRIA

Attempts have been made to correct the conductivity measurements for two or more dissolved gases on the assumption that the correction can be calculated by adding the separate conductivities found when each gas is present alone in the sample. Such a procedure, or any other which assumes an arbitrary ion distribution, neglects the equilibria which must exist between the various ions, as has been pointed out by Watson (74). The problem of calculating these equilibria was rather completely discussed in the paper, "The Calculation of Equilibria in Dilute Water Solutions" (37). The methods described therein are used in this paper.

Due to the high steaming rates of modern boilers, it is probable that equilibrium is never established between the constituents of the steam and the boiler water. It is therefore impossible to say what portions of the more volatile substances, such as CO_2, NH_3, and H_2S, are entrained in the water carried over from the boilers. Fortunately this problem is not important, since the object of the measurement of conductivity is the determination either of the nonvolatile salts which may cause trouble by depositing in superheaters and turbines, or the percentage carry-over of the boiler. If the object is the determination of the nonvolatile salts in the steam, analysis of the steam only may be necessary. If the determination of the percentage carry-over is desired, analysis of both steam and boiler water is necessary.

When analyses are available on steam and boiler water, both the percentage carry-over and the nonvolatile salts may be calculated.

Calculation of Corrections to Conductivity Measurements from Analyses of the Steam Only

The concentration of salts in condensed steam is so low that the conductivity κ can be calculated from the equivalent conductances of the ions at infinite dilution λ_0 by multiplying the concentration of each ion in equivalents per million[10] by λ_0 for the ion in question, adding the results, and multiplying by 10^{-6} as indicated by the equation:

$$\kappa = 10^{-6} \times \Sigma_i \lambda_{0i} \times C_i \ldots\ldots\ldots (1)$$

where:

λ_{0i} = equivalent conductance of ion i at infinite dilution,

C_i = concentration of ion i in equivalents per million[3,4], and

κ = conductivity in mhos per centimeter.

and the sum is taken over all the ions in the solution.

The activity coefficients of all ions in the condensed steam may also be taken as unity since the concentration is very low (that is, activities = concentrations in moles per liter). In order to calculate the correction, it is first necessary to calculate the concentration of all the ions of weak electrolytes present in the steam by the method described in the paper, "The Calculation of Equi-

TABLE I.—ANALYSES OF STEAM AND BOILER WATER.

Steam

Observed Values ($\kappa \cdot 10^6 = 6.0$)	ppm.	millimoles per liter
(*C*) Total CO_2	10	0.23
(*N*) Total NH_3	0.4	0.023

Calculated Values ($\kappa \cdot 10^6$ (corrected for gases) = 1.96, pH = 5.5)	ppm.	millimoles per liter
Free CO_2	8.8	0.20
HCO_3^-	1.4	0.028
NH_4^+	0.40	0.023
$H_2PO_4^-$	0.02	0.0002
Nonvolatile dissolved solids in steam	0.80	......

Boiler Water

Observed Values ($\kappa \cdot 10^6 = 4330$, pH = 11)			Calculated Values					
	ppm.	millimoles per liter	millimoles per liter	epm.	millimoles per liter	epm.	$\lambda_0 \times C$, C = epm.
		(*a*)	(*b*)	(*c*)	(*d*)	(*e*)	(*f*)
Dissolved solids	2092						
Dissolved salts	1933						
P = Total phosphate as PO_4^{---}	50	0.53					
C = Total carbonate as CO_3^{--}	20	0.33					
SO_4^{--}	934	9.73		19.46		19.46	
Cl^-	136	3.83		3.83		3.83	
OH^-			1.008	1.008	1.25	1.25	24.7
HCO_3^-			0.058	0.058	0.032	0.032	1.4
CO_3^{--}			0.272	0.544	0.298	0.595	49.5
HPO_4^{-}			0.482	0.964	0.403	0.806	48.4
PO_4^{---}			0.0482	0.145	0.127	0.381	29.7
NA^+				26.0		26.4	
G_B						−3.06	

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[3] In most of the data on conductivity, concentrations are reported in equivalents or milliequivalents per liter. For the dilute solutions met with in boiler practice, milliequivalents per liter and equivalents per million are practically identical.

[4] Tentative Method for Reporting Results of Analysis of Industrial Waters (D 596 – 40 T), 1940 Supplement to Book of A.S.T.M. Standards, Part III, p. 541.

libria in Dilute Water Solutions" (37). For example, in a water containing NH_3, CO_2, and phosphates, the total concentration of each and the pH must be known. By means of curves constructed from Table IV, the concentration of each of the possible ions involved in the equilibria—namely: NH_4^+, OH^-, H^+, HCO_3^-, CO_3^{--}, $H_2PO_4^-$, HPO_4^{--}, and PO_4^{---}—may be calculated, and from the table of values of λ_0 (Table III) their contribution to the conductivity κ_e is calculated in exactly the same way as indicated in Eq. 1 except that the sum is calculated only for the ions involved in equilibria, as follows:

$$\kappa_e = 10^{-6} \Sigma_e \lambda_{0i} C_i \qquad (2)$$

From these concentrations the value of G[5] in the equation

$$[NH_4^+] + [H^+] - [OH^-] - [HCO_3^-] - 2[CO_3^{--}] - [H_2PO_4^-] - 2[HPO_4^{--}] - 3[PO_4^{---}] = G \qquad (3)$$

can be calculated. (Bracketed symbols indicate concentrations in moles or millimoles per liter.)

It is next necessary to make a decision, in some systematic and acceptable manner, as to which ionic species existed in the solids entrained in the steam (assuming that the steam was completely dried in the superheater) and which were present in the gaseous state. This decision could be based on analyses of boiler water and of the solid deposits found in the system in question. Since the boiler water is always alkaline, all ammonia would be driven off as NH_3 upon drying. One might find by analysis of the deposits that phosphates and a part of the CO_2 were present in the solids as PO_4^{---} and CO_3^{--} ions, and that no free bases existed in the solids. The ions mentioned above must then be combined in such a way as to satisfy these conditions, without changing the value of G, as follows:

$$\begin{aligned} NH_4^+ + OH^- &\rightarrow NH_3\,(gas) + H_2O \qquad (4)\\ H^+ + OH^- &\rightarrow H_2O\\ HPO_4^{--} + OH^- &\rightarrow PO_4^{---} + H_2O\\ H_2PO_4^- + 2OH^- &\rightarrow PO_4^{---} + 2H_2O\\ HCO_3^- + OH^- &\rightarrow CO_3^{--} + H_2O \end{aligned}$$

Upon exhaustion of OH^-, bicarbonate would be decomposed as follows:

$$2HCO_3^- \rightarrow H_2O + CO_2\,(gas) + CO_3^{--} \qquad (5)$$

Since reactions *4* and *5* result in the loss of all ammonia and a part of the CO_2 (all except that which remains as CO_3^{--}), the pH of the sample may change. It would then be necessary, using Table IV, to find a new distribution of the ions satisfying the equilibria with the remaining CO_2 and also maintaining the value of G obtained in Eq. 3. The contribution of the remaining nonvolatile ions involved in the equilibria to the conductivity could then be calculated in exactly the same manner as before (Eq. 2). If we let this contribution equal $\kappa_{e\,(nonvolatile)}$, the corrected conductivity could be calculated thus:

$$\kappa_{corrected} = \kappa_{measured} - (\kappa_e - \kappa_{e(nonvolatile)}) \qquad (6)$$

It will be at once recognized that the above method, while theoretically pos-

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[5] *Source and significance of the quantity G.* The quantity G, the sum of the charges on all the ions involved in equilibria, (Eq. 3) is derived from the equation of electrical neutrality, which states that the sum of the charges on all the ions in a solution is zero. In most cases this equation cannot be used directly, since a complete analysis of the sample is not available. However, it has been assumed that the concentration of each of the ions involved in equilibria is known or can be calculated. The ions in a solution may then be considered in two groups:

Group I.—Ions involved in equilibria (whose concentrations depend on pH.)

Group II.—Ions not involved in equilibria (whose concentrations are independent of pH) such as Na^+ SO_4^{--}, Cl^-, etc. Since the concentrations of these ions are independent of pH, the sum of the charges on them must also be independent of pH.

The equation of electrical neutrality can then be written as follows:

Sum of charges on ions of group I + sum of charges on ions of group II = 0

where, by definition, sum of charges on ions of group I = G. Since the second term of this equation is independent of pH, the first term, G, must also be independent of pH.

The quantity G will not be changed by the solution or removal from solution of a substance, if all the ions of the substance belong either to group I or to group II. For example, solution of CO_2 or NH_3 adds equal amounts of positive and negative charge to the ions of group I and therefore causes no change in G. Similarly, solution of NaCl will not change G, since both ions belong to group II. Solution of NH_4Cl will increase G, since NH_4^+ belongs to group I and Cl^- to group II.

If a change in volume of water occurs, due to evaporation or condensation of pure water, G will vary inversely as the volume, since it represents a charge density. That is, if G_1 and V_1 represent initial values of G and volume, and G_2 and V_2 represent final values, $G_1V_1 = G_2V_2$.

TABLE II.—EQUIVALENT CONDUCTANCE OF ELECTROLYTES AT 25 C.[a]

C (equivalents per liter)...		0.0000	0.0005	0.001	0.005	0.01	0.02	0.05	0.10	Constant a
NaCl	Λ..........	126.45	124.50	123.74	120.65	118.51	115.76	111.06	106.74	1.89
	$\frac{\Lambda_0}{\Lambda}$..........	1	1.016	1.022	1.048	1.067	1.092	1.139	1.185	
	$\kappa \cdot 10^6$.......	0	62.25	123.74	603.25	1185.1	2315.2	5553	10 674	
NaOH	Λ..........	248	246	245	240	237	233	227	221	0.954
	$\frac{\Lambda_0}{\Lambda}$..........	1	1.008	1.012	1.033	1.046	1.064	1.093	1.122	
	$\kappa \cdot 10^6$.......	0	123	245	1200	2370	4660	11 350	22 100	
½ Na_2SO_4	Λ..........	129.9	125.74	124.15	117.15	112.44	106.78	97.75	89.98	4.64
	$\frac{\Lambda_0}{\Lambda}$..........	1	1.033	1.046	1.109	1.155	1.217	1.329	1.444	
	$\kappa \cdot 10^6$.......	0	62.87	124.15	585.75	1124.4	2135.6	4887.5	8988	

[a] From D. A. MacInnis Principles of Electrochemistry," p. 339, Reinhold Publishing Co., New York, N. Y. (1939).

NOTE.—Λ = equivalent conductance of the salt at the concentration indicated.
Λ_0 = equivalent conductance of the salt at infinite dilution.

sible and perhaps occasionally useful for recalculating old data, suffers from several defects.

1. The calculations would be tedious.
2. Too many determinations must be made on the steam. (The measurement of pH alone would cause considerable trouble.)
3. No two chemists could agree on how the ions should be combined.

In view of these serious defects some other method of approach is necessary. Watson (74) has suggested a method which, with slight modification, is suitable when the contaminating gases consist of NH_3 and CO_2. Watson's method consists in carbonating the sample with pure CO_2 until the pH is less than 7, and determining conductivity, free CO_2, and NH_3 on the carbonated sample. The correction is then read from a set of curves calculated from theoretical data, presumably on the assumption that CO_2 and NH_3 represent the only weak electrolytes present in the water. If phosphates, sulfites, or other weak electrolytes are present, their contribution to the conductivity will vary, depending on the final pH of the sample, and the correction will be indefinite to this extent. Watson's method can be improved and this objection eliminated by carbonating to a fixed pH value. The correction then depends only on the ammonia present, and the corrected conductivity corresponds to a definite distribution of the ions of the other weak electrolytes. The corrected conductivity will be, not that of a solution of the nonvolatile electrolytes, but that of a solution of the nonvolatile electrolytes with a standard amount of carbonation. The correction can be calculated as follows: Assuming that we will apply

TABLE III.—IONIC EQUIVALENT CONDUCTANCE AT 25 C.

Ion	λ_0[a]	Ion	λ_0[a]	Ion	λ_0[a]
K^+	73.52[b]	Cl^-	76.34[b]	HS^-	72[g]
Na^+	50.11[b]	NO_3^-	71.44[b]	HSO_3^-	71[f]
H^+	349.82[b]	½ SO_4^{--}	79.8[b]	½ SO_3^{--}	80[d]
NH_4^+	73.4[b]	OH^-	198[b]	$H_2PO_4^-$	29[g]
½ Ca^{++}	59.5[b]	HCO_3^-	44.48[b]	½ HPO_4^{--}	60[e]
½ Mg^{++}	53.06[b]	½ CO_3^{--}	83[c]	⅓ PO_4^{---}	78[e]

[a] λ_0 = ionic equivalent conductance at infinite dilution.

[b] MacInnis, "Principles of Electrochemistry," p. 342. Reinhold Publishing Co., New York, N. Y. (1939).

[c] MacDougall, "Physical Chemistry," p. 477, Macmillan Co., New York, N. Y. (1936). (Value uncertain.)

[d] International Critical Tables, Vol. 6, p. 242, McGraw-Hill Book Co., Inc., New York, N. Y. Rough extrapolation of data on K_2SO_3. (Value uncertain.)

[e] Abbott and Bray, *Journal*, Am. Chemical Soc., Vol. 31, p. 729 (1909). Calculated from 18 C. using $\frac{1}{\lambda}\frac{d\lambda}{dT} = 0.023$ for HPO_4^{--} and 0.025 for PO_4^{---}. Data on $H_2PO_4^-$ from this paper gives λ_0 for $H_2PO_4^-$ somewhat higher than that given above with $\frac{1}{\lambda}\frac{d\lambda}{dT} = 0.0215$. Values for the other phosphate ions were reduced, arbitrarily, to agree with that given for $H_2PO_4^-$, the amount of reduction being proportional to λ_0.

[f] International Critical Tables, Vol. 6, p. 260 (from Λ_0 for ½ H_2SO_3).

[g] International Critical Tables, Vol. 6, p. 260 (from Λ_0 for corresponding acids). Calculated from 18 C. using $\frac{1}{\lambda}\frac{d\lambda}{dT} = 0.0215$.

TABLE IV.—DISTRIBUTION OF IONS OF WEAK ELECTROLYTES AS A FUNCTION OF pH AT 25 C.

pH	H_2O		NH_3		CO_2			H_2S			H_2SO_3*			H_3PO_4			
	$a_{H^+}\cdot 10^3$	$a_{OH^-}\cdot 10^3$	$\frac{a_{NH_4^+}}{a_N}$	$\frac{a_{NH_3}}{a_N}$	$\frac{a_{CO_2}}{a_C}$	$\frac{a_{HCO_3^-}}{a_C}$	$\frac{a_{CO_3^{--}}}{a_C}$	$\frac{a_{H_2S}}{a_S}$	$\frac{a_{HS^-}}{a_S}$	$\frac{a_{S^{--}}}{a_S}$	$\frac{a_{H_2SO_3}}{a_{S^{+4}}}$	$\frac{a_{HSO_3^-}}{a_{S^{+4}}}$	$\frac{a_{SO_3^{--}}}{a_{S^{+4}}}$	$\frac{a_{H_3PO_4}}{a_P}$	$\frac{a_{H_2PO_4^-}}{a_P}$	$\frac{a_{HPO_4^{--}}}{a_P}$	$\frac{a_{PO_4^{---}}}{a_P}$
1	100.0										0.8532	0.1468		0.9302	0.0698		
1.5	31.62										0.6477	0.3523		0.8083	0.1917		
2	10.00				1.0000						0.3676	0.6324		0.5714	0.4286		
2.5	3.162				0.9999	0.0001		1.0000			0.1553	0.8447		0.2966	0.7034	0.0000	
3	1.000				0.9996	0.0004		0.9999	0.0001		0.0549	0.9450	0.00006	0.1176	0.8823	0.0001	
3.5	0.3162				0.9986	0.0014		0.9996	0.0004		0.0180	0.9818	0.0002	0.0405	0.9593	0.0002	
4	0.1000				0.9957	0.0043		0.9988	0.0012		0.0058	0.9936	0.0006	0.0132	0.9862	0.0006	
4.5	0.0316		1.0000		0.9866	0.0134		0.9964	0.0036		0.0018	0.9962	0.0020	0.0042	0.9939	0.0019	
5	0.0100		0.9999	0.0001	0.9587	0.0413		0.9886	0.0114		0.0006	0.9932	0.0062	0.0013	0.9925	0.0062	
5.5	0.0032		0.9998	0.0002	0.8800	0.1200		0.9649	0.0351		0.0002	0.9804	0.0193	0.0004	0.9804	0.0192	
6	0.0010		0.9994	0.0006	0.6988	0.3012	0.0000	0.8969	0.1031		0.0001	0.9412	0.0587	0.0001	0.9415	0.0584	
6.5	0.0003		0.9982	0.0018	0.4232	0.5785	0.0001	0.7333	0.2667		0.0000	0.8352	0.1648	0.0000	0.8361	0.1639	
7	0.0001	0.0001	0.9944	0.0056	0.1883	0.8113	0.0004	0.4651	0.5349			0.6157	0.3842		0.6173	0.3827	
7.5		0.0003	0.9872	0.0173	0.0683	0.9303	0.0014	0.2157	0.7843			0.3363	0.6637		0.3378	0.6622	0.0000
8		0.0010	0.9472	0.0528	0.0226	0.9728	0.0046	0.0800	0.9200			0.1381	0.8619		0.1390	0.8609	0.0001
8.5		0.0032	0.8503	0.1497	0.0072	0.9783	0.0145	0.0268	0.9732			0.0482	0.9518		0.0485	0.9512	0.0003
9		0.0101	0.6423	0.3577	0.0022	0.9530	0.0448	0.0086	0.9914			0.0158	0.9842		0.0159	0.9831	0.0010
9.5		0.0319	0.3622	0.6378	0.0006	0.8701	0.1293	0.0027	0.9973			0.0050	0.9950		0.0051	0.9918	0.0031
10		0.1008	0.1522	0.8478	0.0002	0.6801	0.3197	0.0009	0.9991			0.0016	0.9984		0.0016	0.9885	0.0099
10.5		0.3188	0.0537	0.9463	0.0000	0.4022	0.5978	0.0003	0.9997	0.0000		0.0005	0.9995		0.0005	0.9689	0.0306
11		1.008	0.0176	0.9824		0.1754	0.8246	0.0001	0.9998	0.0001		0.0002	0.9998		0.0001	0.9090	0.0909
11.5		3.188	0.0056	0.9944		0.0630	0.9370	0.0000	0.9997	0.0003		0.00005	0.99995		0.0000	0.7598	0.2402
12		10.08	0.0018	0.9982		0.0208	0.9792		0.9990	0.0010			1.00			0.5000	0.5000
12.5		31.18	0.0006	0.9994		0.0067	0.9933		0.9968	0.0032						0.2403	0.7597
13		100.8	0.0002	0.9998		0.0021	0.9979		0.9901	0.0099						0.0909	0.9091

NOTE.—The symbol *a* as used above indicates activity of a given ion. The fractional activities are best explained by an example: $\frac{a_{CO_2}}{a_C}$ means $\frac{a_{CO_2}}{a_{CO_2} + a_{HCO_3^-} + a_{CO_3^{--}}}$. At infinite dilution the activities become equal to concentrations in moles per liter.

* From $K_1 = 1.72\cdot 10^{-2}$; $K_2 = 6.24\cdot 10^{-8}$. H. V. Tartar and H. H. Garretson, "The Thermodynamic Ionization Constants of Sulfurous Acid at 25 C." *Journal*, Am. Chemical Soc., Vol. 63, p. 808 (1941). All other values from dissociation constants given in W. M. Latimer's "Oxidation Potentials," Prentice Hall, Inc., New York, N. Y. (1938).

a correction just large enough to reduce the corrected conductivity of water containing only CO_2 and NH_3 to zero, and that pH = 6 is satisfactory as the fixed pH value to be attained by carbonation, we find by referring to Table IV, at this pH: $[H^+] = 0.001$ millimoles, $[NH_4^+] = 0.9994\ N$, where N represents the total NH_3 in millimoles per liter. If NH_3 and CO_2 are added to pure water, we must have:

$$[NH_4^+] + [H^+] - [HCO_3^-] = 0 \quad \ldots\ldots(7)$$

Substituting the above values:

$$0.9994\ N + 0.001 - [HCO_3^-] = 0 \quad \ldots(8)$$

Assuming values of N, $[NH_4^+]$ and $[HCO_3^-]$ can be calculated using Eq. 8. The correction to the conductivity may then be calculated by the equation:

$$\text{Conductivity correction (mhos per centimeter at 25 C.)} = 10^{-6}\{\lambda_{NH_4^+} \times (\text{epm. } NH_4^+) + \lambda_{H^+} \times (0.001) + \lambda_{HCO_3^-} \times (\text{epm. } HCO_3^-)\} \quad \ldots(9)$$

CONDUCTIVITY CORRECTION FOR CARBONATION TO pH = 6.

NH_3, ppm.	N	$[NH_4^+]$	$[HCO_3^-]$	Correction, micromhos per cm. at 25 C., $\kappa \times 10^6$
0	0	0	0.001	0.35
0.5	0.0294	0.0294	0.0304	3.85
1	0.0588	0.0588	0.0598	7.33
1.5	0.0883	0.0883	0.0893	10.80
2	0.1177	0.1177	0.1187	14.26

In applying this method, the indicator used should be adjusted to pH = 6 in order that no CO_2 will be required for its titration. The conductivity, corrected by this method, cannot be accurately interpreted in terms of dissolved salts without a knowledge of the composition of the boiler water. However, since the major constituent of most boiler waters is Na_2SO_4, sufficiently accurate results will be obtained by assuming that the corrected conductivity is all due to this salt. With this assumption:

$$\kappa_{\text{corrected}} \times \frac{10^6}{129.9} = Na_2SO_4 \text{ (epm.) (from Table II)}$$

$$\kappa_{\text{corrected}} \times 7.70 \cdot 10^3 = Na_2SO_4 \text{ (epm.)};$$

$$\kappa_{\text{corrected}} \times 5.47 \cdot 10^5 = Na_2SO_4 \text{ (ppm.)}$$

CALCULATION OF THE PERCENTAGE CARRY-OVER AND SOLIDS IN STEAM FROM ANALYSES OF STEAM AND BOILER WATER

A method involving successive approximations may prove useful when analyses of both steam and boiler water are available, or when more than two contaminating gases are present in the steam. This method avoids some of the difficulties of the first method described, since it requires a minimum number of determinations to be made on the steam sample and no assumptions need be made as to the distribution of the possible volatile substances between the entrained solids and the steam. It requires the measurement of conductivity and the concentration of each of the contaminating gases in the steam. In the boiler water; conductivity, pH, and the concentration of each of the weak electrolytes are required.

At first reading, the calculations to be described may appear laborious. A trial will show that the work is less than that required to calculate the results of a complete water analysis from the laboratory determinations. It should be noted also that the most difficult part is the evaluation for the boiler water of κ_B and G_B of Eqs. 11 and 15. In modern boiler plants, the boiler water analysis is held within rather narrow limits. Hence κ_B and G_B need be calculated only infrequently compared to the calculations on the steam. The latter calculations are very simple and easy to make with the help of Tables II and IV.

The method is based upon the following ideas: analysis of steam is difficult due to its extreme dilution; hence the number of determinations to be made on the steam sample should be reduced to a minimum, and the steam analysis calculated, in so far as possible, from the boiler water analysis. To do this it is necessary to have a sufficient number of equations connecting the steam analysis and the boiler analysis to calculate the fraction of boiler water carried over with the steam, the corrections to be applied to the conductivities, and the concentrations of all important substances in the steam. The necessary equations are Eq. 11, relating conductivity of steam and boiler water, Eq. 15, relating G of steam and boiler water, and Eq. 16, relating nonvolatile material in steam and boiler water. These equations are not independent, since they all involve the fraction f of boiler water carried over with the steam. Since the relation between them is not simple, they must be solved by successive approximations.

Referring to Eqs. 1 and 2, it is evident that the contribution of the ions to the conductivity can be separated into two parts such that κ_e represents the contribution to the conductivity

$$\kappa = 10^{-6}\,\Sigma_i\,\lambda_{0i}\,C_i = \kappa_e + \kappa_n \ldots\ldots (10)$$

of all ions whose concentrations are involved in equilibria (that is, are dependent on pH) and κ_n represents the conductivity due to ions not involved in equilibria and is therefore independent of the concentration of the contaminating gases such as CO_2 and NH_3, and also of the concentration of substances like phosphates which may be entrained from the boiler. If the same thing be done for the boiler water, then the fraction of boiler water carried over, f, is:

$$f = \frac{\kappa_{nS}}{\kappa_{nB}} = \frac{\kappa_S - \kappa_{eS}}{\kappa_B - \kappa_{eB}} \ldots\ldots\ldots (11)$$

where the subscripts S and B indicate quantities evaluated for the steam and boiler water, respectively. κ_S is the measured conductivity of the steam. Since boiler water may be too concentrated to permit the assumption that the measured conductivity can be evaluated using the ionic equivalent conductances at infinite dilution, κ_B must be interpreted as the conductivity one would obtain if no interionic forces existed. The conductivity κ_B corrected for interionic forces may be defined by $\kappa_B = 10^{-6}\,\Lambda_0\,C$, whereas the measured conductivity is $\kappa = 10^{-6}\,\Lambda\,C$, whence we obtain:

$$\kappa_B = \frac{\Lambda_0}{\Lambda}\,\kappa \ldots\ldots\ldots\ldots (12)$$

where Λ_0 is the equivalent conductance at infinite dilution and Λ is the equivalent conductance at concentration C. The theory for the conductance of mixed electrolytes is not sufficiently well developed to allow us to predict $\frac{\Lambda_0}{\Lambda}$ for a given solution. If the water analysis is known, the "mixture rule" may be used, as in the example given later in this section. If the analysis is not known, it seems reasonable that this ratio will not differ greatly from that for the predominant salt in the solution (usually Na_2SO_4) when the concentration of this salt is such that its conductivity is the same as that of the boiler water. From the data given in Table II, a plot of κ_B *versus* κ can be made, from which κ_B can be read directly. A plot of $\frac{\Lambda_0}{\Lambda}$ *versus* $\kappa^{\frac{1}{2}}$ is practically a straight line (**41**) whose equation is:

$$\frac{\Lambda_0}{\Lambda} - 1 = a\kappa^{1/2} \text{ (where } a \text{ is a constant for a given electrolyte)}$$

Introducing this into Eq. 12:

$$\kappa_B = (1 + a\kappa^{1/2})\kappa \ldots\ldots\ldots (13)$$

Values of the constant a for various electrolytes are also given in Table II.

The contribution to the conductivity of the ions involved in the equilibria can be easily evaluated using the data of Tables III and IV, and the known pH and concentrations of the boiler water. Since the boiler water is fairly concentrated, it will be necessary to use the activity coefficients of the ions. Hence the ionic strength must at least be approximately known. For a given water supply and treatment, the relative concentrations of the ions in the boiler water vary in a fairly definite manner during the operating run. This fact will allow one to establish a relation of conductivity to ionic strength from previous analyses of the boiler water.

The ion concentrations obtained from this calculation allow the evaluation of the quantity G for the boiler water (see discussion of Eq. 3), where:

G = sum of the charges on all ions involved in equilibria . . . . (14)

If the quantity G is evaluated for both steam and boiler water, then:

$$G_S V_S = G_B V_B$$

And since, at 25 C., the densities of steam and boiler water are nearly identical, the fraction of boiler water in steam is:

$$f = \frac{V_B}{V_S} = \frac{G_S}{G_B} \quad \text{. . . . . (15)}$$

In addition, the nonvolatile materials in the steam may be calculated from those in the boiler water from:

$$f = \frac{\text{nonvolatile material in steam}}{\text{nonvolatile material in boiler water}} \quad \text{. . (16)}$$

The relation 16 will hold for total phosphates and sulfites, and for any other nonvolatile substances.

The procedure for making the calculation is as follows. The denominator of Eq. 11 and G_B are calculated from the boiler water analysis. Assuming a reasonable value for the pH of the steam, the numerator of Eq. 11 is evaluated from the known concentration of the contaminating gases in the steam. An approximate value of f is thus obtained which allows the calculation of G_S and the phosphate, sulfite, etc. in the steam using Eqs. 15 and 16. Making use of graphs constructed from the data of Table IV, a pH is found by trial which satisfies the initial value of G_S. The numerator of Eq. 11 is then recalculated using this new pH value, giving a more accurate value for f. The process is repeated until successive calculations of f produce no further appreciable change. The percentage carry-over is then $f \times 100$.

The procedure is illustrated by the following example, using the water analyses given in Table I.

In order to calculate G_B (Eq. 15) and the denominator of Eq. 11, it is necessary to calculate the ion concentrations existing at equilibrium. Assuming first that the activity coefficients are each unity, and referring to Table IV, we find for pH = 11: $[OH^-] = 1.008$, $[HCO_3^-] = 0.1754C$, $[CO_3^{--}] = 0.8246C$, $[HPO_4^{--}] = 0.9090P$, $[PO_4^{---}] = 0.0909P$. Using these factors, column *b* of Table I is calculated from column *a*, and column *c* calculated by multiplying the quantities in columns *b* or *a* by the valence of the several ions. The concentration of sodium is then found by summing the equivalents per million in column *c* for the negative ions, assuming sodium to be the only metal present. The ionic strength μ is then calculated by the equation

$$\mu = \frac{\Sigma C\nu^2}{2} = 3.663 \times 10^{-2}$$

(In this equation C is the concentration in moles per liter, and ν is the valence of each ion.) The activity coefficients γ

are then calculated using $\log \gamma_i = -0.5066\, \nu_i^2 \sqrt{\mu}$ and found to be:

for monovalent ions, $\gamma_1 = 0.80$
for bivalent ions, $\gamma_2 = 0.41$
for trivalent ions, $\gamma_3 = 0.13$

The corrected ion concentrations are then calculated, using the quantities read from Table IV as follows:

$$[OH^-] = 1.008/\gamma_1$$

$$[HCO_3^-] = \frac{0.1754/\gamma_1}{0.1754/\gamma_1 + 0.8246/\gamma_2}$$

$$[CO_3^{--}] = C - [HCO_3^-]$$

$$[HPO_4^{--}] = \frac{0.9091/\gamma_2}{0.9091/\gamma_2 + 0.0909/\gamma_3}$$

$$[PO_4^{---}] = P - [HPO_4^{--}]$$

The quantities thus obtained are tabulated in column *d*, Table I. Recalculating the sodium concentration from these corrected ion concentrations we find it to be 26.4 (column *e*). (When the pH value is higher than 11, the $[OH^-]$ should be determined in accordance with the A.S.T.M. Tentative Method for Determination of the Hydroxide Ion in Industrial Waters (D 514 - 38 T).[6] Since this result differs but slightly from the result of the initial calculation, and the equation for the activity coefficients is only approximate, the quantities entered in column *d* and *e* are taken as the correct concentrations. From column *e*, G_B is found to equal -3.06. The contribution of each ion involved in equilibria to the conductivity, entered in column *f* of Table I, is calculated by multiplying the values from column *e* by the proper λ_0 values from Table III.

The quantity κ_B, the conductivity corrected for interionic attraction, may be calculated in several ways, depending on the completeness of analysis of the boiler water. If the sulfate and chloride concentrations are unknown, perhaps the best assumption would be that the boiler water behaves like a solution of the predominant salt (Na_2SO_4) having the same conductivity. From a plot of $\frac{\Lambda_0}{\Lambda}$ *versus* κ for Na_2SO_4 (Table II), $\frac{\Lambda_0}{\Lambda}$ is found to be 1.31.

If the sulfate and chloride are known, a better value is obtained by using the "mixture rule" (**2, 67**) that is, by taking the weighted mean of the Λ values for Na_2SO_4 and NaCl, each read from a plot of Λ *versus* C, at the equivalent concentration of the boiler water. At 26 epm., $\Lambda = 104.3$ for Na_2SO_4 and 114.5 for NaCl. The weighted mean $\frac{\Lambda_0}{\Lambda} = \frac{4 \times 129.9 + 1 \times 126.45}{4 \times 104.3 + 1 \times 114.5} = 1.22$ assuming all uni-univalent salts behave like NaCl and all uni-bivalent salts behave like Na_2SO_4. Using this value:

$$\kappa_B = \kappa \times 1.22 = 4330 \times 1.22 = 5280$$

The denominator of Eq. 11 is then:

$$5280 - (\text{sum of values given in column } f, \text{ Table I}) = 5280 - 154 = 5130$$

The numerator of Eq. 11 must next be calculated. Assuming as a first approximation that the pH of the steam is determined solely by the CO_2 and NH_3, G_S must be approximately zero. (By the equation of electrical neutrality, G_S would be exactly zero if the entrainment were zero.) From plots of the data of Table IV we obtain for the steam:

$$[H^+] + [NH_4^+] - [HCO_3^-] = G_S \text{ (approximate)}$$

at pH 5 , $0.0101 + 0.023 - 0.0413 \times 0.23 = 0.0236$
at pH 5.5, $0.0032 + 0.023 - 0.12 \times 0.23 = -0.0014$
at pH 5.4, $0.004 + 0.023 - 0.10 \times 0.23 = 0.004$

Using the values at pH 5.5, the numerator of Eq. 11 is found to be:

$$6.0 - (350 \times 0.0032 + 73.4 \times 0.023 + 44.5 \times 0.0276) = 1.96$$

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[6] 1939 Book of A.S.T.M. Standards, Part III, p. 1111.

The first approximation to f (Eq. 11) is then:

$$f = \frac{1.96}{5130} = 3.82 \times 10^{-4}$$

Whence, using Equation 15:

$$G_S = f \times G_B = 3.82 \times 10^{-4} \times (-3.06) = -0.0012$$

and phosphate in the steam, using Eq. 16 is:

$$P_S = f \times P_B = 3.82 \times 10^{-4} \times 0.53 = 0.0002$$

where P_S and P_B represent the total phosphate in steam and boiler water, respectively. The value of G_S just found must agree with that used for calculating f, from which it was first necessary to omit the phosphate. Recalculating this as follows:

$$[H^+] + [NH_4^+] - [HCO_3^-] - [H_2PO_4^-] = G_S$$

$$\text{At pH 5.5,}\quad 0.0032 + 0.023 - 0.12 \times 0.23 - 0.98 \times 0.0002 = -0.0016.$$

The values for G_S (that is, -0.0012 and -0.0016) agree as well as could be expected, considering the possible errors in analysis of both steam and boiler water. If reasonable agreement is not obtained on first trial, the process is repeated until successive values of G_S agree. If the change produced in G_S by a change of pH of 0.1 is greater than the difference between successive values of G_S, the agreement is satisfactory. Again using Eq. 16:

$$\text{Dissolved solids in steam} = f \times \text{dissolved solids in boiler water} = 3.82 \times 10^{-4} \times 2092 = 0.80 \text{ ppm.}$$

If desired, the complete analysis of the steam may be calculated. Some of the calculated values are summarized in the steam analysis of Table I.

The data given in Tables II and IV have not been presented in curve form because of the difficulty and expense of reproducing curves of sufficient accuracy to be useful. It is felt that more useful and readable plots can be made by the reader. It is very likely that this method can be simplified in specific cases. Due to the lack of sufficiently complete data in the literature, the author has been unable to try the method under a sufficiently wide variety of conditions to decide when simplification is possible. It is hoped that these conditions will be revealed by trial of the method.

The reader may wish to consult one or more of the following books for a detailed discussion of the methods and theory of conductance measurements:

M. Dole, "Experimental and Theoretical Electrochemistry," McGraw-Hill Book Co., New York, N. Y. (1935).

D. A. MacInnis, "Principles of Electrochemistry," Reinhold Publishing Corp., New York, N. Y. (1939).

S. Glasstone, "Electrochemistry of Solutions," D. Van Nostrand Co., New York, N. Y. (1937).

H. J. Creighton, "Electrochemistry," Vol. I. John Wiley and Sons, Inc., New York, N. Y. (1935).

JOINT DISCUSSION

Experimental Methods of Determining Conductivity Corrections for Dissolved Gases In Steam Condensate

By S. F. Whirl and W. A. Lower

Calculation of Corrections to Conductivity Measurements for Dissolved Gases

By D. S. McKinney

Mr. **Everett P. Partridge**.[1]—One basic assumption has been applied but not explicitly stated by Mr. Whirl. He has assumed that, when ammonia and carbon dioxide are distilled from the steam condensate and collected in water free of dissolved salts, the conductivity measured in the distillate is identical with the increment in conductivity produced by these gases in the actual steam condensate containing dissolved salts from carry-over. However, there will be at least a theoretical difference, the magnitude of which will depend upon the extent to which hydroxide or phosphate has been introduced into the steam condensate by carry-over of boiler water. In most cases this difference will be negligible, but it seems wise to emphasize whenever possible the fact that the effect of ammonia and carbon dioxide, separately or together, upon conductivity is not fixed, but depends upon the pH of the solution in which they are present.

Anyone who will take the time to study Mr. McKinney's most thorough paper must stand indebted to him. Here is outlined in complete detail what can be done to obtain from observed conductivity readings a theoretically correct indication of the dissolved solids in a sample of steam condensate. I should like to point out only that, for ordinary work, much of the calculation necessary in the rigorous application of his procedure on pages 1292 to 1295 may be avoided.

In Eq. 11, the fraction of boiler water in the steam is expressed as

$$f = \frac{K_S - K_{eS}}{K_B - K_{eB}}$$

The second term in both the numerator and the denominator is really a calculated correction to be subtracted from the corresponding first term derived from actual measurement. Using the values in the example calculated in the paper from the data in Table I,

$$f = \frac{K_S - K_{eS}}{K_B - K_{eB}} = \frac{6.0 - 4.04}{\underbrace{(4330 \times 1.22)}_{5280} - 154} = 3.82 \times 10^{-4}$$

it is evident that the correction of 4.04 for the effect of dissolved NH_3 and CO_2 in the steam must be applied, but that the correction of 154 for the effect of ions involved in equilibria dependent upon pH may be neglected in comparison with the value of 5280 for the boiler water calculated to infinite dilution. In fact, omission of this correction changes the calculated value for dissolved salts in the steam only from 0.80 to 0.78 ppm.

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[1] Director of Research, Hall Laboratories, Inc., Pittsburgh, Pa.

Under the condition of very slight contamination of the steam assumed in the problem, the effect of any shift in the phosphate equilibrium in the steam condensate with pH is almost infinitesimal. The numerator of Eq. 11 was therefore obtained by applying to the measured conductivity of the steam condensate the correction calculated from the determined values for NH_3 and CO_2. To save time in calculating the correction, it is possible to use the curves of Watson[2] reproduced in the accompanying Fig. 1. For example, Mr. McKinney has assumed in his example steam containing 0.4 ppm. NH_3 and 10 ppm. total CO_2. The "free" CO_2, or undissociated carbonic acid corresponding to the latter value was calculated by him to be 8.8 ppm. From Watson's curves, the correction to be subtracted from the observed conductivity would be read as 4.1, which compares well with the value of 4.04 calculated by McKinney.

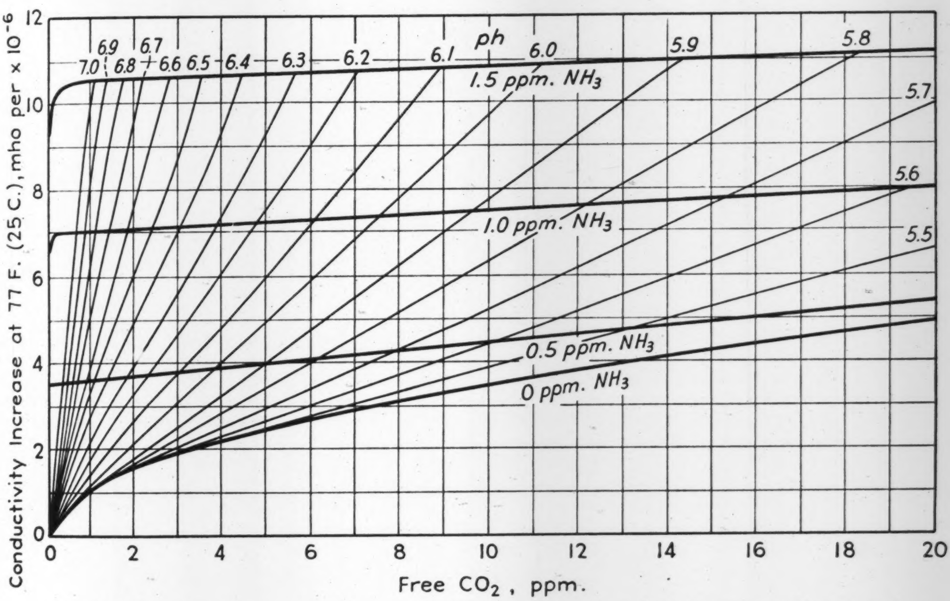

FIG. 1.—Effect of Ammonia and Carbon Dioxide on Conductivity of Steam Condensate.

It has been noted above that, in the example given by Mr. McKinney, the correction term in the denominator of Eq. 11 may be neglected with little loss in accuracy. The first term is calculated very simply by multiplying the observed conductivity of the boiler water by the factor necessary to convert it to a hypothetical state of infinite dilution. This factor may be obtained, as pointed out by the author, either as a first approximation by assuming that the boiler water is Na_2SO_4, or by applying the law of mixtures.

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[2] When originally developed, and when presented as discussion of the paper by W. B. Gurney, M. C. Schwartz and T. E. Crossan, "Determination of Purity of Steam by the Electrolytic-Conductivity Method," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 62, pp. 728–733 (1940), see pp. 732–733, these curves were based upon an assumed value of 70 for the conductivity of HCO_3^- ion. They have now been revised, using the value of 44.5 given by D. A. MacInnes, "The Principles of Electrochemistry," p. 342, Reinhold Publishing Corp., New York, N. Y. (1939), which was brought to our attention by Mr. McKinney. A reference net of calculated pH values has also been added.

Copies of these curves on 8½ by 11-in. coordinate paper are available on request from Hall Laboratories, Inc., 300 Ross St., Pittsburgh, Pa.

It was possible to discard the correction term in the denominator in the example cited, because the concentrations of ions involved in equilibria dependent upon pH, that is, phosphate and carbonate, were low relative to those of ions whose equilibria are practically independent of pH, that is, sulfate and chloride. If this relation were reversed, as it might be in a central-station boiler with evaporated make-up, the correction term might become so large in comparison with the first term that it should not be neglected.

As mentioned in the companion paper in this symposium by Mr. Kaufman,[3] even the most precisely measured and precisely corrected value for the conductivity of steam condensate does not yield a direct answer to the practical questions of the boiler operator, but must instead be interpreted in the light of experience in the particular plant concerned. While expressing gratitude to Mr. McKinney for his most thorough analysis of the correction problem, the writer feels that the simplified procedure suggested above will be adequate for most studies in power plants. The extreme case in which it might be desirable to follow completely the rigorous treatment given in the paper would be provided by a boiler with uncontaminated condensate return and uncontaminated distilled make-up in which only a phosphate salt was used for internal conditioning, since carry-over would then provide chiefly ions whose equilibria were dependent upon pH.

I wonder whether Mr. Whirl could give us a summary of the lack of correlation between steam contamination and turbine deposits. I think that might be interesting.

MR. J. B. ROMER.[4]—The determination of steam quality has presented several problems. Conductivity measurements have always been hampered by the corrections necessary for dissolved gases. The authors are to be congratulated on their work. As has been brought out at different times, ammonia and carbon dioxide should be eliminated, if at all possible, in order to reduce the correction factors to the lowest possible point.

It was the experience of Mr. Rummel, and it has been our experience, that careful work will produce results indicating that the gas corrections are correct—certainly within the limits of the entire procedure.

Any contribution that tends to increase the accuracy of the correction, or to produce a method whereby the gases can be completely eliminated, is a worthwhile contribution to this whole problem.

Steam containing one-quarter part of solids per million parts represents a degree of purity that is rarely obtained in any industrial process. This is certainly true in any large-scale production. Modern turbines handle many millions of pounds of steam in the course of a single run and if any appreciable percentage of the solids in steam adheres to any one stage of the turbine, loss in turbine efficiency quickly becomes a serious item and for this reason a high-grade steam is very important to the powerhouse industry.

MR. M. C. SCHWARTZ.[5]—It should be pointed out that the measurement of steam purity will give varying results, depending upon the method used. For example, gravimetric analysis of the evaporated residue from a condensed steam sample shows a large proportion of the residue to be iron. An analysis of the same condensed steam sample by electrolytic conductivity measurement does not indicate the presence of iron in solution. The significance of the presence of iron in the condensed steam remains to be determined. However, it is interesting to note that the iron content of the condensed steam sample seems to be independent of the nature of the steam sample, that is, whether saturated or superheated. The iron appears to originate from the boiler water, which itself is oxygen free.

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[3] See p. 1326.

[4] Chief Chemist, The Babcock & Wilcox Co., Barberton, Ohio.

[5] Assistant Professor of Chemical Engineering, and Research Associate in Water Technology, Louisiana State University, Baton Rouge, La.

Another point worth considering in the problem of determining steam purity is the correlation between the material found as impurities in the steam and the nature and amount of the materials that become adherent in the steam system beyond the boiler. The amounts of materials adhering appear to be less than would be expected from consideration of steam purity and steam flows. Certain constituents in the steam, such as silica, become more adherent than other more soluble constituents.

The relationship between the impurities carried over into the steam from the boiler water to the chemical composition of the boiler water itself does not appear to be proportional.

All these considerations which are complex in actual practice deserve the attention of all workers in this interesting field.

MR. M. D. BAKER[6] *(by letter)*.—The importance of low carry-over of dissolved solids in steam cannot be overemphasized, as the increased demand on the generating capacities of all power stations requires that units remain in service for prolonged periods. The result is that carry-over that was once permissible because it was removed by washing each time the unit was taken out of service cannot now be tolerated, as the shut-down periods are further apart and the accumulation on the blades may cause a large loss in efficiency and output.

Topping units with periods of months and sometimes over a year of continuous operation demands clean steam for their operation. A unit that uses 1,000,000 lb. of steam per hr., if that steam contains 0.25 ppm. of solids, has delivered to it 6 lbs. of solids each 24 hr. The retention on the blades of a small percentage of this material will quickly affect the unit. Also, the retention of solids in the superheater may cause tube failures and subsequent removal of a boiler from service.

These facts make it imperative that the quality of the steam be known. To obtain the information, a rapid, accurate, and dependable method for analyzing the steam is required. Also, the saturated steam must be sampled at a sufficient number of points so that a true cross-section of all the steam delivered to the superheater will be known. Representative superheated steam samples by themselves will tell the quality of the steam delivered to a turbine but will not give any indication of the deposition in the superheater. As mentioned by the authors, the conductivity method is the most rapid and is best adapted for a continuous flow determination, but it is accompanied with the error caused by the presence of dissolved gases. The correction for these gases must be made to determine the actual solids that are carried over in the steam. The elimination of the gases by the controlled concentration of a condensed steam sample and measuring the conductivity of the solids remaining is a simple and practical method that has proved satisfactory. This method gives one correction for all the dissolved gases but does not attempt to analyze the gases.

Gas corrections made by concentrating the condensed steam samples obtained during actual steam quality tests on boilers do not agree with the data as presented in Table I of the paper by Whirl and Lower, which tabulates the results of laboratory experiments regarding the elimination of ammonia. Numerous tests made by concentrating a sample to one fifth its original volume gave zero ammonia by direct Nesslerization. To obtain these results it was necessary to avoid contamination by reabsorption of gases during cooling of the sample. Care had to be taken in the preparation of the Nessler's reagent and in the actual testing for ammonia. The greatest error was found to be in the quality of the Nessler's reagent used. Zero ammonia was obtained on concentrated steam samples that originally contained 0.4 ppm. ammonia and 1.0 ppm. dissolved solids. The ammonia determination on the concentrated samples was made only to determine if all the dissolved gases originally present had been eliminated. The assumption was that if the ammonia was eliminated, the other gases would also be absent.

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[6] Chief Chemist, West Penn Power Co., Springdale Station, Pittsburgh, Pa.

The procedure used for routine testing at Springdale Station is to collect samples simultaneously from the eight sampling points across the boiler drum. At the same time conductivity readings are made in a flow cell that discharges into the sample flask. One liter of each sample is placed in a two-liter Erlenmeyer flask and then evaporated to approximately 200 ml. The concentrate is then carefully cooled, and the conductance is determined. The volume of concentrate is measured, after which the ammonia test is made to determine whether all dissolved gases have been removed. The solids in the original sample are then calculated by dividing the amount of solids in the concentrate by the number of concentrations made by evaporation.

THE CHAIRMAN (*Mr. R. E. Hall*[7]).—I have a question I should like to ask Mr. Whirl. I noticed that in the table he gave, he reported analyses for CO_2 of 0.5 and 1 part per million, and so on. Can we get accuracies of that sort with certainty?

MR. F. R. OWENS[8] (*by letter*).—It is my opinion that the method as proposed by McKinney will serve as an excellent referee method, but, as a guide in daily operations, I question its applicability.

It is essential, especially in high-pressure operations, to resort to a check on steam quality which is continuous; hence obviously any batch method of determining the mineral content of the steam will be found to be inadequate. It has been our experience that by resorting to suitable means for degasification of samples of condensed steam prior to determining the conductivity, a method results whereby a continuous check is maintained on the steam quality and at the same time correction factors for carbon dioxide and ammonia become negligible, because with adequate reduction of the partial pressure of these two gases and sufficient time for their removal, the correction factor will be fairly constant, at least for practical purposes.

From the operating viewpoint, accuracy in the determination may be sacrificed for speed and continuity. It is nonessential whether the mineral content of the steam is 0.42 ppm. or 0.50 ppm. It is essential that the operator have at his disposal a means which quickly and continuously reveals radical increases in the amount of entrainment, since he can then resort to corrective measures without delay.

A method such as the author outlines can be used advantageously for occasional checks on the continuous method of maintaining a check on steam quality.

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[7] Director, Hall Laboratories, Inc., Pittsburgh, Pa.

[8] Cyrus Wm. Rice and Co., Inc., Pittsburgh, Crafton, Pa.

MESSRS. S. F. WHIRL[9] and W. A. LOWER[9] (*authors' closure, by letter*).—In developing the method outlined in this paper for correcting conductivity measurements for the presence of dissolved gases, the authors sought to eliminate the two principal sources of error involved in other laboratory procedures: namely, errors in analyses and those due to a disregard of the effect of the gases on each other and on the solution. This was accomplished by the elimination of all chemical tests and the determination of one correction factor which represents the combined effects of all gases.

As explained by Mr. Partridge, in those cases where the pH of the steam sample differs from that of the distillate, the measured correction will not be theoretically accurate. As he points out, the differences will usually be negligible, however. The magnitude of the correction will, in general, far overshadow the minute amounts of alkaline chemicals normally encountered in steam; in fact, this correction is usually greater than that of the conductivity due to all of the salts.

Again the authors wish to emphasize that the analytical procedures in common use are a possible source of errors of considerable magnitude in any method based on their use. This is particularly true at low concentrations, especially for "free" carbon dioxide. The data in Table III were presented solely for the purpose of comparing the performance of the proposed method with that of other procedures which involve analytical testing.

Mr. Baker's remarks concerning the quality of the Nessler reagent in the determination of ammonia are most timely. The temperamental behavior of this solution is fully recognized. An investigation in the authors' laboratory showed that sensitivity is a function of both the chemicals used and the age of the solution. However, the use of freshly prepared standards tends to compensate for its variable sensitivity. A possible explanation of Mr. Baker's diverse results may be his use of permanent standards (**3**).[10] On the basis of the authors' experience, it is difficult to understand the values of 0.00 ppm. of ammonia which he has reported.

In answer to Mr. Partridge's question concerning the correlation of steam condensates and steam turbine deposits, the amount of solid matter in the steam is only one of the controlling factors. Of major interest are the amount and location of those chemicals which "stick and stay stuck." Briefly, the other determining variables are: the nature of the solids, temperature, pressure, velocity, pH of the steam, and the design and operation of the turbine. In an Edison Electric Institute report (**48a**) based on the analysis of data from 42 operating stations, the importance of operation was stressed very strongly. It was stated that "Stations with swinging loads or with frequent idle periods, by virtue of the washing effect of the moist steam, are not troubled appreciably, and then, in most cases, only with small amounts of insoluble deposits." It was also concluded from this survey that the solid content of the boiler water is not an index of the composition of the turbine deposits.

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[9] Chief Chemist, and Chemical Laboratory Supervisor, respectively, Duquesne Light Co., Pittsburgh, Pa.

[10] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

THE DEGASIFICATION OF STEAM SAMPLES FOR CONDUCTIVITY TESTS

By P. B. Place[1]

Synopsis

This paper is a summary of the methods and apparatus available for the elimination from steam samples, of gaseous impurities that affect conductivity measurements. The principles involved in the various methods are briefly discussed and the apparatus as developed by various investigators is described.

Complete elimination of gases depends on the continuous removal of the gases from above the liquid sample and on the time allowed for liberation of the residual gases from the liquid under low-differential partial-pressure conditions.

In their present state of development, continuous flow degasifiers will reduce carbon dioxide to an amount that constitutes a negligible conductivity error for commercial testing. Ammonia, being more soluble and having a very high conductivity, is much more difficult to reduce to negligible amounts, and it is usually necessary to make conductivity corrections for the residual ammonia left in the degassed sample. At least one investigator claims complete elimination of both carbon dioxide and ammonia, and with proper control of time, temperature, scavenging, and surface conditions, it appears that complete elimination is possible.

One of the problems in the development of the conductivity method for testing steam purity has been the correction for, or elimination of, acid and basic gases in the steam which yield ionized compounds in solution that contribute to the conductivity of the sample but which yield no solid residue on evaporation. Although such gases as CO_2, NH_3, H_2S, and SO_2 constitute an impurity in the steam, they are not classified as such in a commercial sense because they yield no solid residue that will deposit in superheater tubes or on turbine blades.

There are obviously two methods of approach to this problem. The amount of gaseous impurity in the sample may be determined and a suitable correction applied to the conductivity of the sample, or the gaseous impurity may be eliminated from the sample before the conductivity of the latter is measured.

The determination of a suitable correction for the gaseous impurities is difficult, although much progress has been made in this direction. Not only is the conductivity of some of these gases much greater than the conductivity of the neutral salts normally present in the steam, but the amount of gases may vary considerably because the gas carry-over from the boiler is not a function of the moisture carry-over but rather a function of the rate of gas liberation in the boiler.

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[1] Combustion Engineering Co., Inc., New York, N. Y.

In view of these difficulties, it would appear to be easier and more desirable to eliminate the gases from the sample before the conductivity tests are made and thus avoid making any corrections. Other papers in this symposium deal with the determination of, and correction for, gaseous impurities and this discussion will deal wholly with methods that have been developed for elimination of the gases from the steam sample.

THEORY

Gases are held in solution in condensed steam in two ways. The gases may be in simple solution by virtue of their partial pressure, or they may combine in solution for form ionized compounds. Gases in simple solution are relatively easy to eliminate and do not form ionized compounds that affect conductivity tests. Acid and basic gases combine with water or with each other to form ionized compounds that do affect conductivity and such gases are more difficult to remove than gases in simple solution.

The elimination of gases from simple solution is the reverse of absorption, which follows Henry's law. This law states that at any constant temperature, the weight of gas dissolved by a definite volume of liquid is proportional to the partial pressure of the gas above the liquid. The greater the partial pressure imposed on the liquid, the more gas there is in solution when equilibrium conditions are established. Conversely, the lower the partial pressure of gas above the liquid, the less gas there is in solution under equilibrium conditions.

All methods of degasification are primarily aimed at reduction of partial pressure. In degasification, the gas is liberated from the liquid to approach the partial pressure corresponding to the equilibrium conditions. For complete elimination of the gas, it is necessary to continuously destroy this equilibrium by continuously removing the liberated gas from the space above the liquid and to allow time for the residual gas to liberate itself under conditions of a constantly decreasing differential partial pressure.

As in all reversible reactions governed by the law of mass action, the rate at which equilibrium is reestablished by further liberation of gas will decrease as the concentration of dissolved gas decreases. It will also decrease as the differential partial pressure between gas in solution and liberated gas, decreases. It is evident that for complete elimination of gases from solution, not only must the liberated gas be completely removed from the space above the liquid, but time must be allowed for the liberation of residual gas under constantly decreasing, low-differential, partial-pressure conditions.

A resistance to the rate of elimination is the rate of diffusion through the liquid and through the gas film at the surface of the liquid. The rate of diffusion through the interface is a controlling factor in the elimination of gases from liquids in the quiescent state. Boiling, bubbling inert gas through the liquid, shaking, or otherwise disturbing the interface films, contributes greatly to speeding up the rate of transfer in an unbalanced equilibrium and degasification may be materially hastened by increasing the temperature of the solvent, by reduction in the total pressure above the liquid, or by use of scavenging carriers such as steam or an inert gas which sweeps the liberated gas out of the liquid and away from the space above the liquid.

The usual undesirable gases found in steam are CO_2 and NH_3. Gases other than these are seldom found and this discussion will be limited to these two. These two gases, in solution, unite with water or with each other to form one or more unstable salts. An equilibrium is set up between the amount of gas above the liquid, the amount in simple solution, and the amount in chemical combination. The distribution of the gas in this equilibrium depends on temperature, concentration, pH value and other factors. McKinney (**37**)[2] and Amorosi and McDermet (**1**) have discussed this equilibrium and described methods for calculation of the distribution in dilute solution.

Here again degasification depends largely on reduction in partial pressure. Reduction or elimination of the gas in simple solution shifts the unstable equilibrium to the left and results in reduction of the ionized portion of the gas. Although such factors as temperature and partial pressure can be controlled to favor the elimination of the gas in simple solution and reduction in the ionized portion, the pH value of the sample cannot be controlled by the addition of chemicals without making the sample useless for steam purity tests. Elimination therefore depends not only on the continuous removal of gas above the liquid, but also on maintenance of conditions of temperature, surface, scavenging, and time to favor the shift in distribution of gas from ionized form to simple solution.

It is generally accepted that CO_2 may be easily reduced to an amount that does not constitute an appreciable conductivity error in steam purity tests, but that NH_3 is much more difficult to reduce to a similarly negligible amount. The reason for this is probably threefold. First, CO_2 is less soluble than NH_3 and for the same concentration it has a proportionately larger partial pressure to work on. Second, the conductivity of NH_3 is about ten times that of CO_2 and it is necessary to reduce the NH_3 concentration to a much lower value to give a similar conductivity error. Third, CO_2 cannot be detected in small amounts with the same accuracy as NH_3 and it is relatively easy to obtain negative tests that indicate complete elimination of this gas. If tests for CO_2 were as accurate as for NH_3, conclusions as to the relative ease of complete elimination of these two gases might be different.

There may be some question as to whether a very soluble and high conductivity gas such as NH_3 can be sufficiently eliminated in continuous flow degasifiers or by simple distillation to reduce its conductivity error to a negligible value. The elimination of an acid gas such as CO_2 is favored by a low pH value and the elimination of a basic gas such as NH_3 is favored by a high pH value. Small amounts of carry-over from an alkaline boiler water should favor rather than inhibit elimination of NH_3 and there appears to be no good reason why complete elimination cannot be obtained if the various conditions of temperature, time, scavenging, and surface are suitably controlled.

CLASSIFICATION OF METHODS OF DEGASIFICATION

The various methods in use for the elimination of gaseous impurities in steam samples may be classified as follows:

1. Continuous sample methods:
    (*a*) Elimination by boiling.
    (*b*) Elimination by evacuation.
    (*c*) Elimination by aeration.
2. Batch sample methods:
    (*a*) Elimination by distillation.

In the continuous methods, a continuous flow of sample is maintained through the degasifier, in which a com-

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

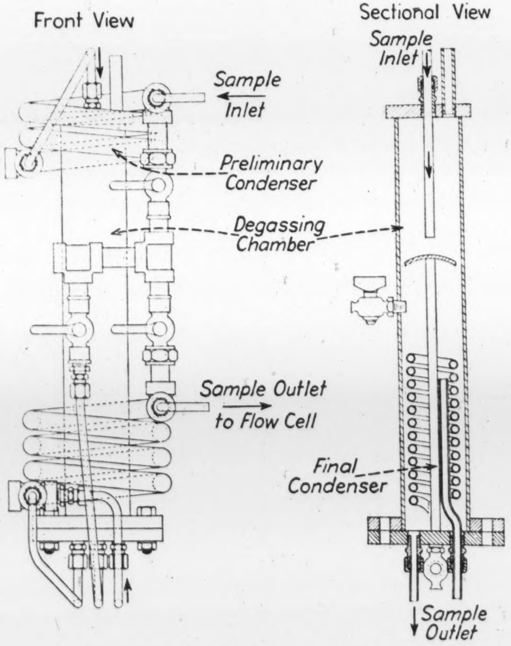
Front View
Sectional View
Sample Inlet
Sample Inlet
Preliminary Condenser
Degassing Chamber
Sample Outlet to Flow Cell
Final Condenser
Sample Outlet

FIG. 1.—Rummel Degasifier.

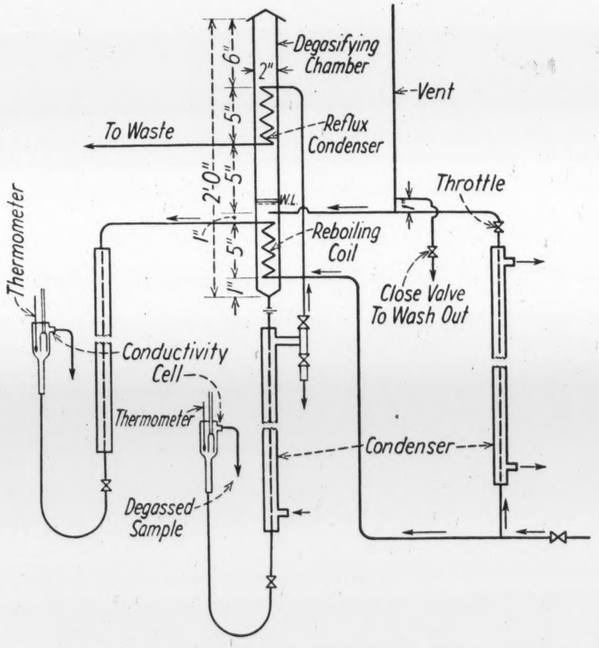
Degasifying Chamber
2"
6"
5"
5"
5"
1"
2'-0"
1"
Vent
To Waste
Reflux Condenser
W.L.
Throttle
Reboiling Coil
Close Valve To Wash Out
Thermometer
Conductivity Cell
Thermometer
Condenser
Degassed Sample

FIG. 2.—Gurney, Swartz, and Crossan Degasifier.

plete and continuous elimination of gas is attempted. The degasifier is followed by a conductivity flow-type cell for measurement of the conductivity of the degassed sample.

In batch methods, steam condensate samples are collected and tested individually. Distillation in some form is the basic process in these methods and the time required for such tests is so long that only occasional results can be obtained. The value of batch method tests is chiefly in checking continuous flow conductivity records and in checking composite samples collected over a period of time. When the amount of gas in the steam is low and fairly constant, the conductivity of the steam may be determined continuously without degasification and the correction for the gases determined occasionally by a batch method test. Similarly, if the continuous flow degasifier does not give complete elimination of the gases, the residual gas in the sample may be determined by occasional batch tests and suitable corrections applied to the continuous conductivity record of the partially degassed sample.

Application of Continuous Elimination by Boiling:

Soon after the conductivity method had been applied to steam condensate testing, Rummel (53) developed a continuous flow degasifier. The apparatus, modified somewhat from the original design, is shown in Fig. 1. The steam sample is first partially condensed in the external cooling coil located at the top of the apparatus. The hot mixture of steam and condensate is then admitted to the degassing chamber in which is located a second cooling coil and to which is attached a vent. By careful control of the flow of cooling water to the preliminary and final condensers, the condensate which collects in the degassing chamber is maintained at the boiling point and the liberated gases, together with some scavenging steam, are vented to the atmosphere. The gas-free condensate is drawn off continuously from the bottom of the chamber, cooled in an outside cooling coil, and its conductivity measured in a flow-type cell.

A somewhat different design of degasifier of the boiling type was developed by Gurney, Schwartz, and Crossan (20) and is shown in Fig. 2. In this apparatus, the condensed steam delivered to the degassing chamber is reboiled by a steam heated coil submerged in the condensate. A reflux condenser located in the upper part of the chamber prevents excessive loss of steam vapor but allows venting of the liberated gases. By controlling the steam flow to the reboiling coil and the cooling water supply to the reflux condenser, the condensate in the chamber is kept at the boiling point and the liberated gases escape thru the vent. The steam used in the reboiling coil is part of the steam sample and the conductivity of the condensate from this coil may be determined to give data on the undegassed steam.

The above apparatus will eliminate all but a negligible amount of CO_2 and reduce the NH_3 concentration, but residual NH_3 should be determined separately and suitable corrections applied to the continuous conductivity record. A typical residual of 0.14 ppm. of nitrogen in the form of ammonia, is reported by Gurney as having a conductivity correction of 1.12 micromhos.

Straub and Nelson (63) recently reported the development of the continuous degasifier shown in Fig. 3. This apparatus is described as making use of the principle of boiling a sample of condensed steam from which the gases have been removed, to furnish gas-free steam, which in turn is used to scrub the gases from the incoming sample. The principle feature of this apparatus is the use of a scrubbing tower or stripping column imposed over the boiling chamber. This tower contains a large amount of surface in the form of perforated plates over which the condensed steam sample flows in a thin film while being degassed. A reflux condenser located above the stripping column prevents excessive loss of steam but permits venting of the liberated gases and a small amount of scavenging steam. The steam sample is first admitted under pressure to the reboiling coil located in the bottom of the degassing chamber, where it is partially condensed in reboiling the collected condensate. The partially condensed sample is next completely condensed and cooled in the upper external cooling coil. Part of this condensate is passed through a flow cell for determination of the conductivity of the undegassed sample and a controlled portion of the remainder is admitted to the degassing chamber through an orifice and preheating coil. The preheated sample is distributed and trickled down over the perforated plates and is degassed by the steam rising from the boiling condensate in the bottom of the chamber. With this apparatus, under controlled conditions of flow, complete elimination of both CO_2 and NH_3 is claimed.

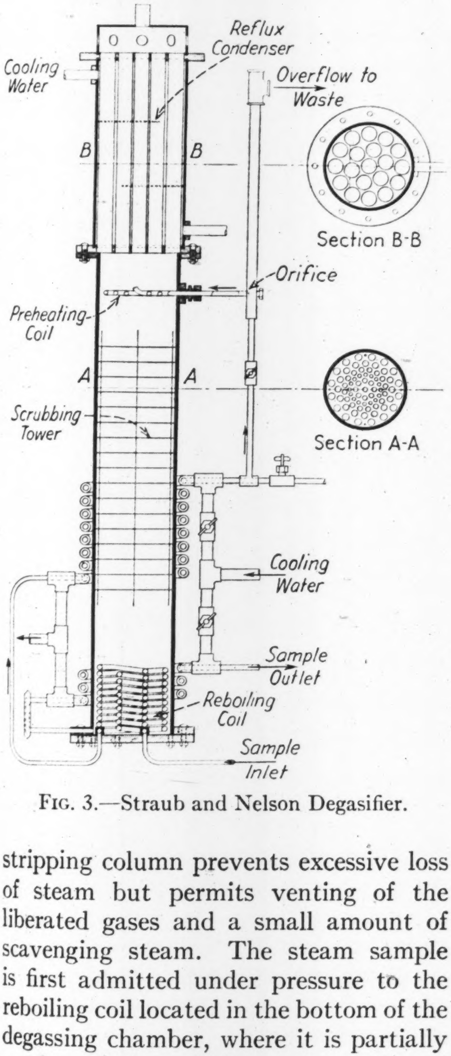

FIG. 3.—Straub and Nelson Degasifier.

Figure 4 shows a somewhat similar apparatus developed by the Elliott Co. (12). It utilizes the scrubbing tower principle, the incoming sample being distributed and trickled over a series of plates located in the steam space above the boiling degassed sample. An unusual feature of this equipment is the use of electric heating elements for boiling the condensate. Although primarily designed for removal and collection of gases in steam condensate, the apparatus is suitable for degasification of steam samples for conductivity tests, provided the electric heating elements do not introduce undesirable contamination. An advantage of the electric heating system is its adaptability to automatic control for maintenance of the proper operating conditions. The sample drawn off for conductivity test is cooled to a suitable temperature in an outside cooling coil not shown in the diagram.

The degasifier shown in Fig. 5 is classified as of the boiling type, but has the additional feature of atomization. Although designed primarily for the separation and collection of hydrogen and other gases from steam, it is applicable to degasification of the steam samples for conductivity tests and has

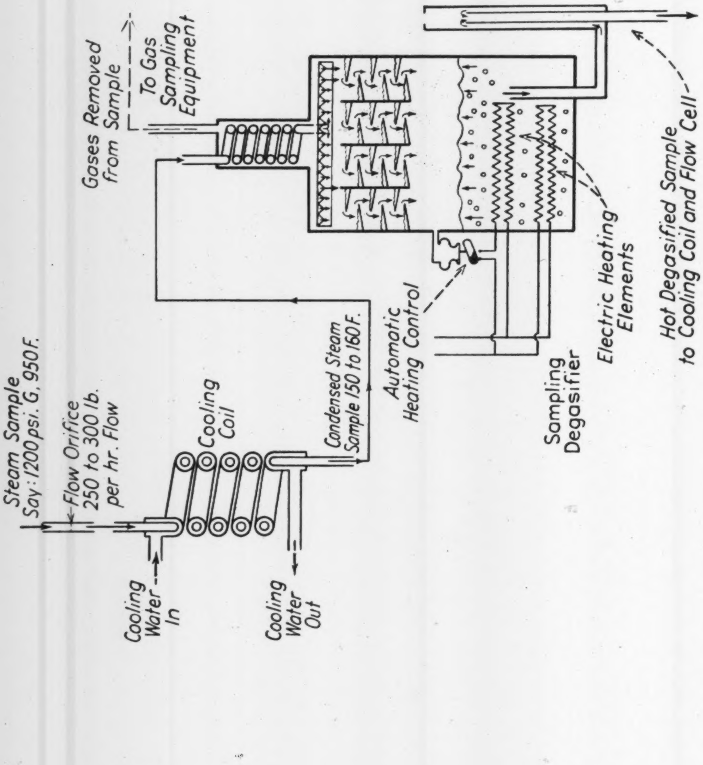
Steam Sample
Say: 1200 psi. G, 950 F.
Flow Orifice 250 to 300 lb. per hr. Flow
Cooling Coil
Cooling Water In
Cooling Water Out
Condensed Steam Sample 150 to 160 F.
Automatic Heating Control
Sampling Degasifier
Electric Heating Elements
Hot Degasified Sample to Cooling Coil and Flow Cell
Gases Removed from Sample
To Gas Sampling Equipment

FIG. 4.—Elliot Co. Deaerator.

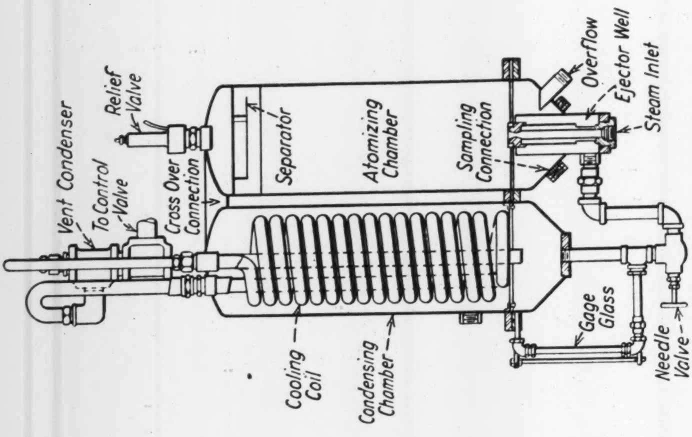
Vent Condenser
To Control Valve
Relief Valve
Cross Over Connection
Separator
Atomizing Chamber
Sampling Connection
Overflow
Ejector Well
Steam Inlet
Needle Valve
Gage Glass
Cooling Coil
Condensing Chamber

FIG. 5.—Joos Atomizing Degasifier.

been described by Joos (29). The apparatus consists of two chambers, one of which is the condensing chamber and the other the atomizing chamber. Steam is admitted under pressure through an orifice located in the bottom of the atomizing chamber. The condensate which collects in the bottom of this chamber is atomized and boiled by the incoming steam and the steam vapor and liberated gases are swept over into the condensing chamber in which the steam is recondensed and from which the gases are vented. The condensed steam is recirculated to the atomizing chamber and the atomization continuously repeated. The hot, degassed condensate is drawn off from the bottom of the atomizing chamber and cooled in an outside cooling coil before passing through a flow-type cell for conductivity measurement. No data on removal of CO_2 and NH_3 are available, but the repetition of the atomizing cycle favors efficient elimination of these gases.

Application of Continuous Elimination by Evacuation:

Attacking the problem of degasification from a different angle, Powell (46), and Powell, Bacon, McChesney, and Henry (47) developed the apparatus shown in Fig. 6. In this apparatus, the steam sample is condensed under reduced pressure and discharged into a flow type cell which is evacuated to a pressure approaching the vapor pressure of water at the sample temperature. The high vacuum, aided by the scavenging effect of small amounts of water vapor drawn off through the evacuating element, maintains a practically gas-free atmosphere in the system. The temperature in the evacuated flow cell is normally below 80 F. and suitable for direct conductivity measurement without fur-

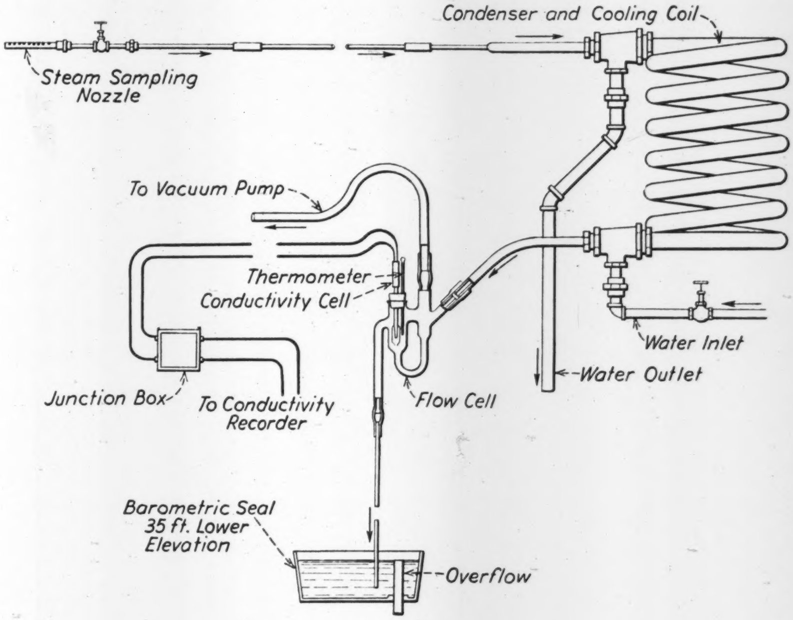
Condenser and Cooling Coil
Steam Sampling Nozzle
To Vacuum Pump
Thermometer
Conductivity Cell
Water Inlet
Water Outlet
Junction Box
To Conductivity Recorder
Flow Cell
Barometric Seal 35 ft. Lower Elevation
Overflow

FIG. 6.—Powell Vacuum Degasifier.

ther cooling. The overflow from the cell is through a barometric water leg which serves as a seal. With this apparatus, large amounts of CO_2 have been reduced continuously to negligible residuals. Ammonia is reduced but complete elimination is not claimed. Residual NH_3 is determined and suitable corrections applied to the continuous conductivity record.

Application of Continuous Elimination by Aeration:

An example of aeration applied to a continuous flow method of degasification is described by Powell, Bacon, McChesney, and Henry (**47**). The apparatus is shown in Fig. 7. The condensed steam is atomized under pressure by a small spray nozzle and the resulting mist is aerated by an induced current of clean air. The mist is condensed on the walls of the aeration chamber and collects in a flow cell in which its conductivity is measured. The air, together with the liberated gases, escapes through a vent. This method is effective in reducing large and variable amounts of CO_2 to small and fairly constant amounts for which suitable corrections can be applied. No data is available on elimination of NH_3.

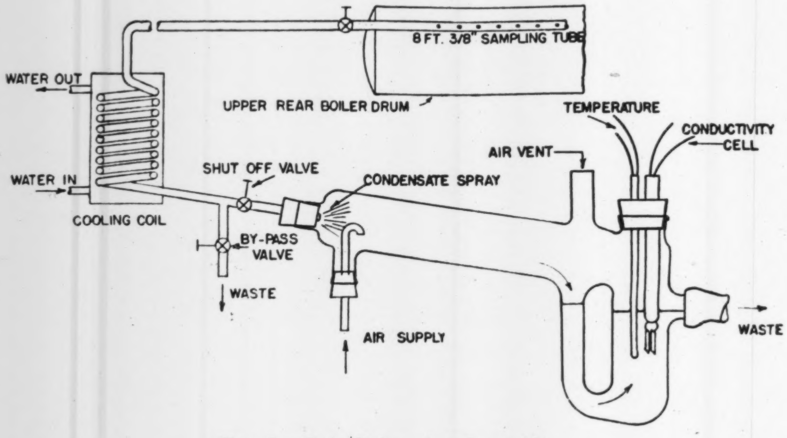

FIG. 7.—Powell Aerating Degasifier.

Application of Batch Elimination by Distillation:

Practically all batch methods involve boiling and distillation, although aeration and evacuation are as applicable to batch methods as to continuous methods. In some cases, a considerable portion of the sample is boiled away with the liberated gases and the nonvolatile impurities determined in the concentrated residue. In other cases, the object of the test is to distill off and collect the liberated gases to determine a suitable correction for conductivity data. Since methods involving the determination of gas corrections are dealt with in another paper,[3] the only method discussed here will be one in which the primary object is the determination of the nonvolatile impurities in the concentrated residue after the volatile impurities have been distilled off.

The author has described such a distillation method (**45**), using the apparatus

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[3] S. F. Whirl and W. A. Lower, "Experimental Methods of Determining Conductivity Corrections for Dissolved Gases in Steam Condensate," see p. 1277.

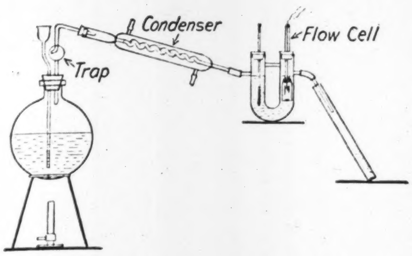

FIG. 8.—Apparatus for Batch Distillation Test.

shown in Fig. 8. A 1- to 2-liter sample is distilled and the progress of gas evolution noted by conductivity and titration tests on the distillate. When these tests indicate no further evolution of gas, the conductivity and volume of the cooled residue in the still is measured and the purity of the original sample calculated by proportion.

A typical distillation test is shown in Fig. 9. A 2300-ml. sample containing 8.5 ppm. of CO_2 and having a conductivity of 5.5 micromho, is concentrated by distillation to a 300 ml. residue having a conductivity of 2.9 micromho. The large rectangle and crosshatched square at the bottom of Fig. 9 represent, respectively, the conductivity of the sample and the proportion of that conductivity due to nonvolatile impurities.

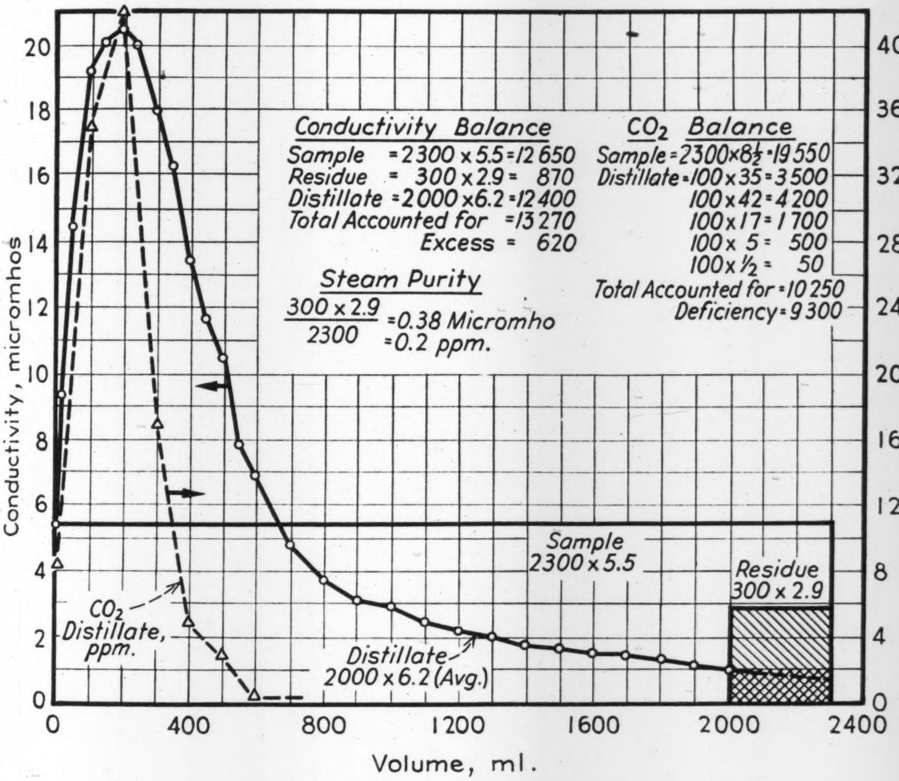

FIG. 9.—Typical Distillation Test.

Since the slope of the conductivity curve at the end of the distillation indicates that gases are still being eliminated, the curve may be extrapolated to complete distillation and the double hatched area subtracted as a correction. Although such correction may be questioned, it serves to counteract high results due to residual NH_3 or contamination. In this case, the corrected steam conductivity would be

$$\frac{300 \times (2.9 - 0.8)}{2300} = 0.27 \text{ micromho}$$

It has been found that CO_2 can be eliminated by this method but that last traces of NH_3 are found in the concentrated residues. The amount of residual NH_3 has been reported as varying from 0.03 ppm. to 0.20 ppm. (57). These residuals are, however, in the concentrated samples and when calculated to actual correction on the original sample, vary from 0.02 to 0.36 micromho. These corrected values are not excessively large and normally will not exceed 0.2 micromho.

For accurate work it is necessary to determine these residuals and make correction for them, but in the average run of commercial testing it is probable that the accuracy of sampling has not kept pace with the accuracy of the purity determination even when these corrections are not applied. There is little justification for reporting steam purities of less than one half part per million, closer than the nearest tenth of a part. In most cases, the presence of insoluble carry-over equals if not exceeds the correction made for residual ammonia.

SUMMARY

Only acid and basic gases which combine chemically in solution to give ionized compounds affect conductivity measurements. An equilibrium exists between the amount of gas in combination, the amount in simple solution, and the amount above the liquid as partial pressure. Elimination is based primarily on reduction in partial pressure which destroys the equilibrium and favors reduction of the amount of gas in ionized form. The more soluble the gas is, the less the amount of partial pressure to work on and the more completely must the partial pressure be eliminated to reduce the dissolved gas to negligible amounts.

Methods of elimination may be applied to continuous or batch samples. Such standard processes of elimination as boiling, distilling, evacuating, aerating, and atomizing have been applied. A number of continuous degasifiers are available for commercial use. Distillation methods on batch samples are used for laboratory control and for checking composite samples.

It has been found easy to reduce CO_2 to negligible amounts but difficult to similarly reduce NH_3. Although small residuals constitute a large error in the conductivity of very pure steam samples, they may not constitute a serious error in commercial testing in which the methods of sampling are often questionable.

Acknowledgment:

In closing, I wish to express my appreciation for the cooperation of the several investigators listed herein, who kindly furnished me with diagrams and subject matter for this paper.

DISCUSSION

MR. M. C. SCHWARTZ.[1]—I should like to ask whether one should be able to get out all the ammonia, or even all the carbon dioxide, from a sample of condensed steam from a degasifier?

From the theory involving the pH of the sample, it seems to me that it should not be possible. I should appreciate it if some of the authors of the papers in this symposium would answer this question.

MR. P. B. PLACE.[2]—Probably the direct answer to this question is no, but if we rephrase the question to read "Can the ammonia and carbon dioxide be reduced in a degasifier to residuals that do not represent a serious error in steam purity determinations?", then there might be two answers. The research chemist may still say no but the practical test engineer could, I believe, say yes.

Proper degasification will reduce the gases to a practically constant minimum and eliminate variations and fluctuations in conductivity that might be misinterpreted.

The operating man wants not only a low steam conductivity but a constant one and he will judge the purity by the rate at which his turbine blades foul. The present degasifier is primarily designed as a piece of operating equipment which, with further development, may approach but never reach the standard of a research apparatus.

It is likely that the errors introduced by steam sampling and presence of insoluble and un-ionized carryover exceed the errors due to small gas residuals.

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[1] Assistant Professor of Chemical Engineering, and Research Associate in Water Technology, Louisiana State University, Baton Rouge, La.

[2] Combustion Engineering Co., Inc., New York, N. Y.

A NEW TYPE OF CONDUCTIVITY APPARATUS FOR USE WITH BOILER WATERS AND STEAM SAMPLES

By A. R. Mumford[1]

Synopsis

A new type of conductivity apparatus for use with boiler waters or steam samples is described. Gas contamination is prevented in the case of steam samples by condensing in contact with the vapor phase and slight venting. Degasification of water samples is secured by flashing and venting the vapor phase. The cell is designed for use at full steam pressure and saturation temperatures. Measurement of conductivities at elevated temperatures as compared to "standard" temperatures reduced hydroxyl ion correction to a negligible amount.

The apparatus is rugged, uses little cooling water, may be installed adjacent to the sampling point, makes possible simple recovery of the condensate, makes possible continuous reading without appreciable time lag, and is undamaged by failure of cooling water.

The conductivity apparatus described in this paper was developed for the purpose of simplifying and improving the making of conductivity measurements as an aid to power plant operation. The apparatus, however, is such that it can be used as a research instrument, particularly at the higher temperatures and pressures.

In Fig. 1 is shown one form of the apparatus used in the development to date; Fig. 2 is a photograph of the same apparatus. It may be considered as a condensing cylinder draining into a cell chamber. The sample inlet is at the left end of the condensing cylinder, the condensate drain at the bottom of the cell chamber, the vent at the upper right of the cell chamber, and a pressure gage connection at the top of the cell chamber. The condensing cylinder is jacketed for cooling purposes and partly filled with stainless steel balls. The vapor condenses on the balls and the condensate drains to the bottom of the cylinder, washing any solids along with it. The uncondensed vapor fills the space above the draining condensate and above the electrode cup. The vent allows the noncondensible gases and a predetermined portion of the vapor to escape through capillary tubing to the atmosphere.

The condensate drains through a spout into a standpipe and thence to the electrode cup from which it overflows through a weir. A small hole is provided at the bottom of the solution cup to permit draining of the cup for checking the open circuit resistance in place and to remove continuously any sludge that may settle from highly contaminated solutions. The rate of condensation is kept high enough to cause a change of

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[1] Senior Research Associate, Consolidated Edison Company of New York, Inc., New York, N. Y.

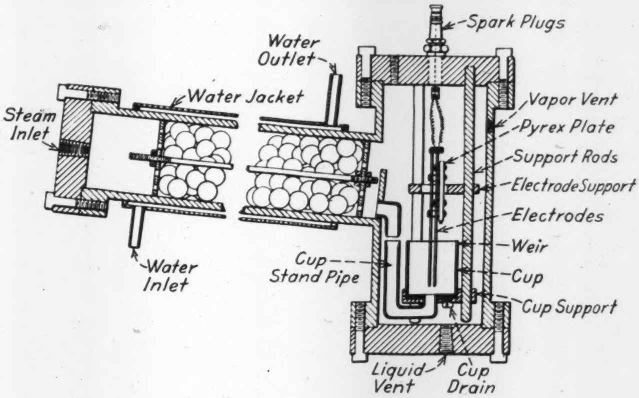
Spark Plugs
Water Outlet
Water Jacket
Steam Inlet
Vapor Vent
Pyrex Plate
Support Rods
Electrode Support
Electrodes
Weir
Cup
Cup Support
Water Inlet
Cup
Stand Pipe
Liquid Vent
Cup Drain

FIG. 1.—High-Pressure Conductivity Apparatus for Vapors or Flashed Liquids.

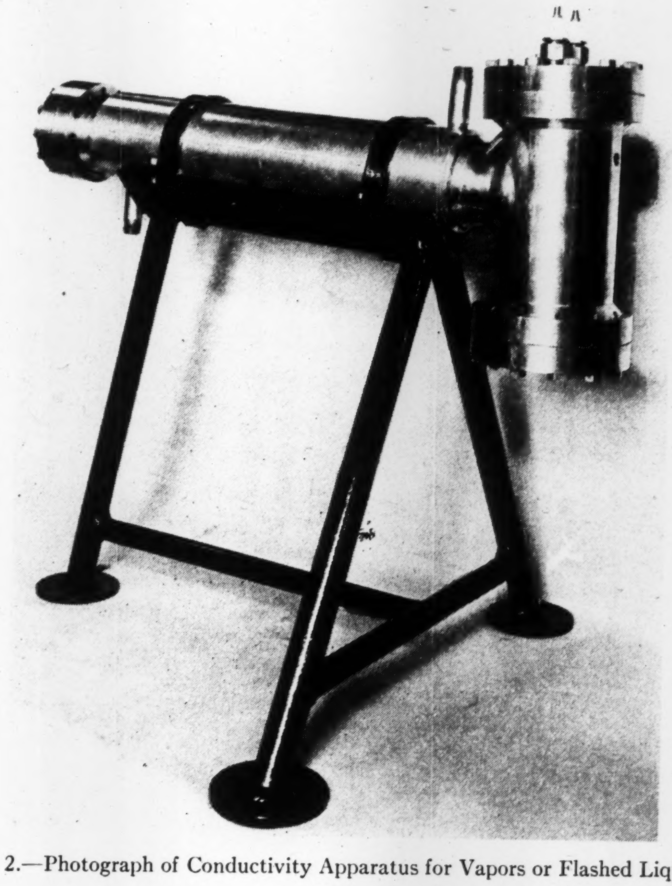

FIG. 2.—Photograph of Conductivity Apparatus for Vapors or Flashed Liquids.

condensate in the electrode cup several times a minute.

The electrode and cup are supported from the cell chamber cover and may be removed as a unit. The electrode wires are brought out through special porcelain spark plugs, which are made with the central wire of stainless steel. All material exposed to vapor or condensate is made of 18 per cent chromium, 8 per cent nickel steel with the exception of the pyrex spacing plate and the lead wires. We expect to use stabilized 18-8 steel in future models for use at high pressures and temperatures.

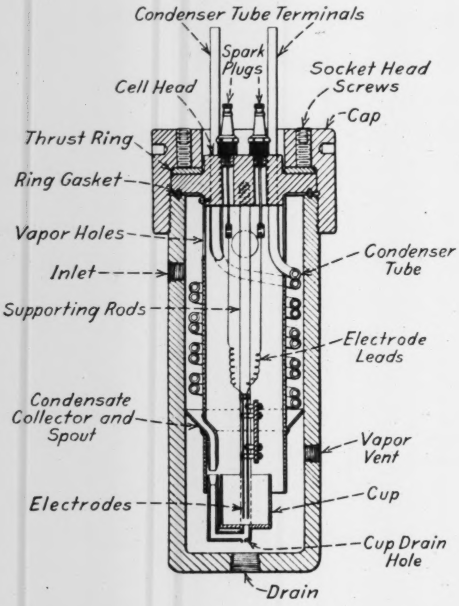

FIG. 3.—New Arrangement of Conductivity Apparatus for Vapors or Liquids.

In Fig. 3 is shown another form of the apparatus designed for use at 1600 psi. and capable of withstanding temperatures of 900 F. The apparatus consists of a tubular body open at the top and provided with three welded pipe connections: an inlet at the left, a drain at the bottom, and a vent at the right. The open end of the body is closed by the cell head, which is sealed against the body by a lock-head construction in which the bolts are under compression. All remaining equipment is fastened to the cell head and is removable with the head as a unit. A tube, partly open near the top and extending part way down the body, divides the space into an annular chamber and an inner core space. Within the annular chamber is placed the cooling coil on which the condensate forms and from which it drains into the electrode cup. Within the central core is placed the electrodes, the spacers, and the support for both electrodes and cup.

TABLE I.—COMPARISON OF PLATINUM AND STAINLESS STEEL ELECTRODES IN MEASUREMENTS OF CONDUCTIVITY OF SOLUTIONS OF KCl.

Concentration	Bridge Multiplier	Measured Resistance, ohms		Ratio of Steel to Platinum
		Platinum	Stainless Steel	
0.1 *N*	1	7.9	8.2	1.04
0.1 *N*	10	8.4	9.0	1.07
0.01 *N*	1	34	34	1.00
0.01 *N*	10	34.3	35.4	1.03
0.001 *N*	10	260	260	1.00
0.001 *N*	100	261	259	0.99
0.001 *N*	1 000	268	268	1.00
0.0001 *N*	100	1 910	1 880	0.98
0.0001 *N*	1 000	1 876	1 867	1.00
0.0001 *N*	10 000	1 960	1 940	0.99
Distilled water	1 000	24 000	24 000	1.00
Distilled water	10 000	24 700	24 700	1.00

Figure 4 shows a form of the apparatus designed for use solely with hot condensates or boiler waters. The vapor type apparatus can, of course, be used for this service, but if a permanent installation for condensates only is desired, the construction can be lighter and the cooling jacket eliminated. There are two principal differences in the arrangement of the apparatus. The hot liquid enters the flash chamber through a perforated tube and the balls are replaced by screens. Where the same apparatus may be called upon to function for both vapors and liquids, it is necessary to use the condensing type. A photograph of the disassembled apparatus is shown in Fig. 5.

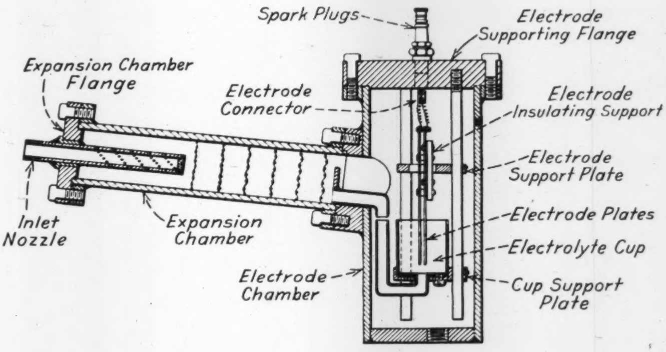
Spark Plugs
Electrode Supporting Flange
Expansion Chamber Flange
Electrode Connector
Electrode Insulating Support
Electrode Support Plate
Electrode Plates
Electrolyte Cup
Cup Support Plate
Inlet Nozzle
Expansion Chamber
Electrode Chamber

FIG. 4.—High-Pressure Conductivity Apparatus for Flashed Liquids Only.

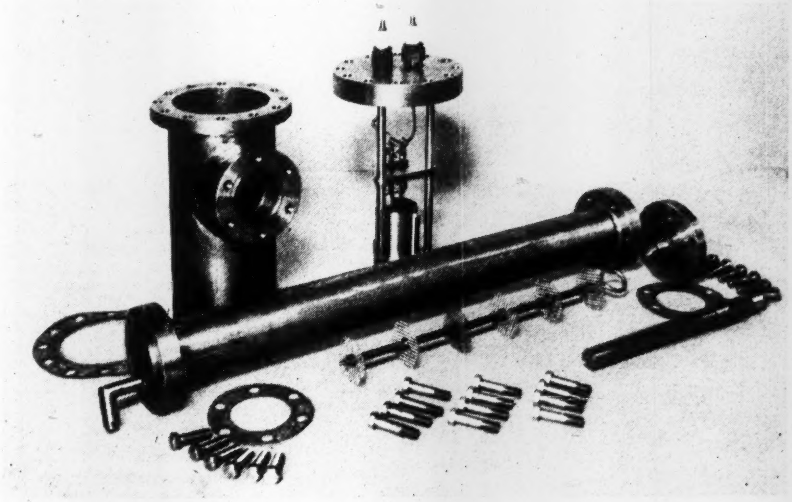

FIG. 5.—Photograph of Disassembled Conductivity Apparatus for Flashed Liquids Only.

Vapor entering the apparatus is cooled to its saturation point and part of it is condensed in contact with the vapor phase. Under such conditions the solubility of gaseous contaminants will be very low according to Hall and Mumford (**24**)[2] and the partial pressure of the gaseous contaminants may be kept low by adequate venting. In none of many chemical analyses does the author recollect finding either oxygen or carbon dioxide in condensate formed from and in full contact with the contaminated vapor phase when not subcooled. In this manner, the solution of the contaminating gases is prevented.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

The solids carried with the vapor are washed down into the electrode cup by the draining condensate.

According to T. J. Finnegan (private communication) stainless steel compares favorably with platinum as an electrode material at low concentrations of dissolved material. The results of a test are given in Table I.

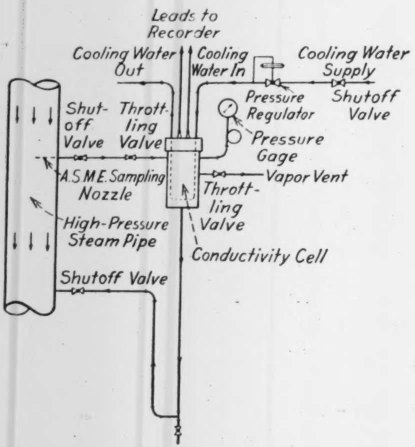
Leads to Recorder
Cooling Water Out
Cooling Water In
Cooling Water Supply
Shut-off Valve
Throttling Valve
Pressure Regulator
Shutoff Valve
Pressure Gage
Vapor Vent
A.S.M.E. Sampling Nozzle
Throttling Valve
High-Pressure Steam Pipe
Conductivity Cell
Shutoff Valve

FIG. 6.—Schematic Diagram of Installation on High-Pressure Steam Main.

The surface of the plate of the electrodes of each set was 6 sq. cm. The distance between the plates was 2.5 mm. in the case of the platinum electrodes and 3 mm. in the case of the stainless steel electrodes. The measured cell constants were 0.0345 for the platinum electrodes and 0.0340 for the stainless steel in 0.0001 *N* KCl. The electrodes were all in the same solution at each strength. The nitrogen was bubbled through each solution to keep it free of CO_2. A 60-cycle a-c. bridge was used at 5 v. The comparison was made at laboratory temperature of 25 C.

Stainless steel electrodes have been used in the studies to date. In addition to stage heater condensates, studies have been made on boiler waters with concentrations up to 300 or 400 ppm. without difficulty.

Hot condensate or boiler water is reduced in pressure before being allowed to enter the apparatus. The reduction in pressure is sufficiently great to provide flashing to remove, with the vapor, any small quantities of noncondensible gases that may have been dissolved because of high partial pressures at some point in the system. Cooling water is allowed to flow through the coil or jacket in quantities sufficient to maintain the desired chamber pressure. Venting is accomplished as before.

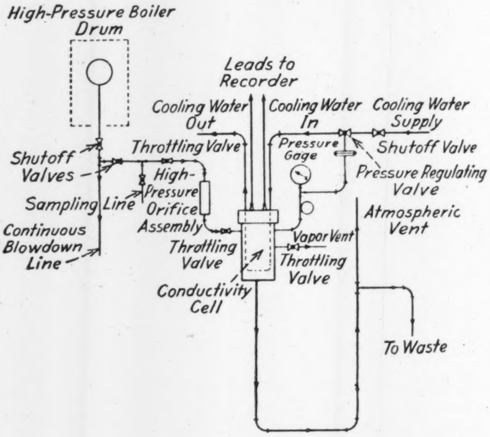
High-Pressure Boiler Drum
Leads to Recorder
Cooling Water Out
Cooling Water In
Cooling Water Supply
Shutoff Valves
Throttling Valve
Pressure Gage
Shutoff Valve
High-Pressure Orifice Assembly
Pressure Regulating Valve
Sampling Line
Atmospheric Vent
Continuous Blowdown Line
Vapor Vent
Throttling Valve
Throttling Valve
Conductivity Cell
To Waste

FIG. 7.—Schematic Diagram of Installation on High-Pressure Blowdown Line.

It is obvious that the condensate passing through the cup will be more concentrated than an unvented sample in proportion to the amount vented. The quantity can, however, be readily fixed by a capillary tube resistance and the total quantity of the sample can also be controlled. The correction therefore is arithmetical for all practical purposes.

Should it be desired to know the specific conductivity, it will be necessary to extend the work of Noyes (40) to boiler water salines at elevated temperatures and to relate the conductivity found

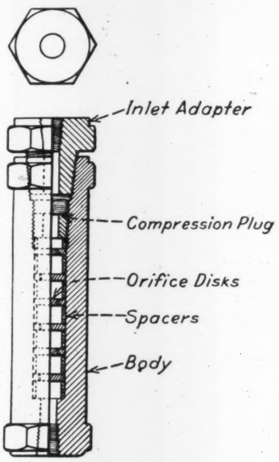
Inlet Adapter
Compression Plug
Orifice Disks
Spacers
Body

FIG. 8.—Multiple Orifice Assembly.

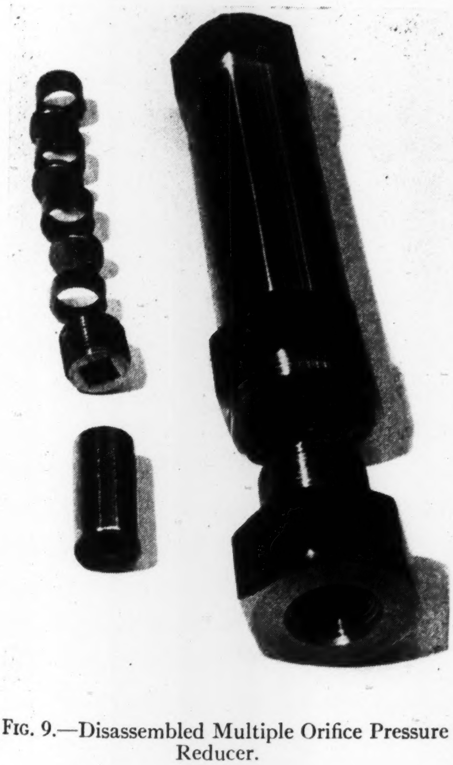

FIG. 9.—Disassembled Multiple Orifice Pressure Reducer.

at saturation temperature to that at "standard" or laboratory temperature. However, operators of generating stations are seldom concerned with specific conductivity but are always concerned with that value of conductivity which may constitute an upper limit of solids content that will unduly interfere with operation.

Kraus (**33**) points out that at elevated temperatures the conductivity of H^+ and OH^- ions relative to acetate ion decreases rapidly. At 300 C., the ratio is of the order of twice the conductivity of acetate ion. The effect of temperature on relative conductivity may be greater when the salt ions are those of boiler water, according to data now being collected. This suggests that the measurement of conductivity at elevated saturation temperatures may reduce the hydroxyl correction to a negligible value and thus eliminate a tedious step in such determinations.

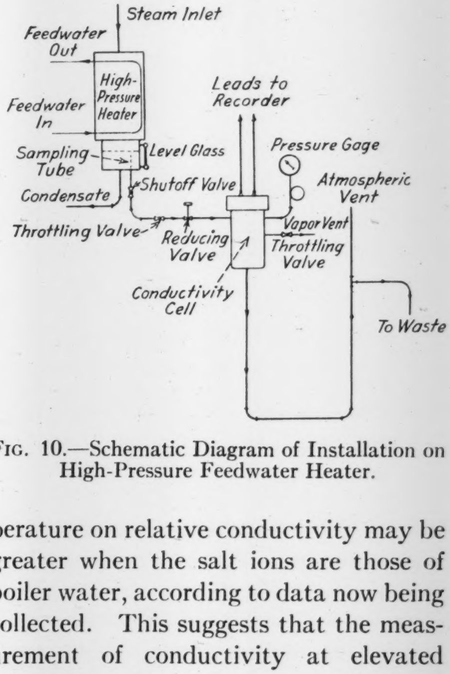
Steam Inlet
Feedwater Out
High-Pressure Heater
Feedwater In
Leads to Recorder
Sampling Tube
Level Glass
Pressure Gage
Shutoff Valve
Atmospheric Vent
Condensate
Throttling Valve
Reducing Valve
Vapor Vent
Throttling Valve
Conductivity Cell
To Waste

FIG. 10.—Schematic Diagram of Installation on High-Pressure Feedwater Heater.

In Fig. 6 is shown, schematically, the manner in which one form of the apparatus was installed for study of its operation on high-pressure steam. The cell shown is the single cylinder type detailed in Fig. 3 and differs from the original cell in that the cooling water connections must be broken before the head assembly can be removed. The pressure regulator on the cooling water is necessary when the cold-water pressure is variable. The throttling valve on the vent represents a fixed resistance. If the pressure at the source of sampling varies in a substantial manner, it is advisable to arrange to operate the apparatus at a constant pressure just below the minimum source pressure by using a pressure-reducing system. The seal dimensions on the condensate discharge are designed to provide a sufficient water leg to offset the slight pressure drop through the apparatus and thus permit the return of the condensate to the superheated steam line when the line pressure does not vary substantially. The seal is so valved that the condensate may be discharged to waste.

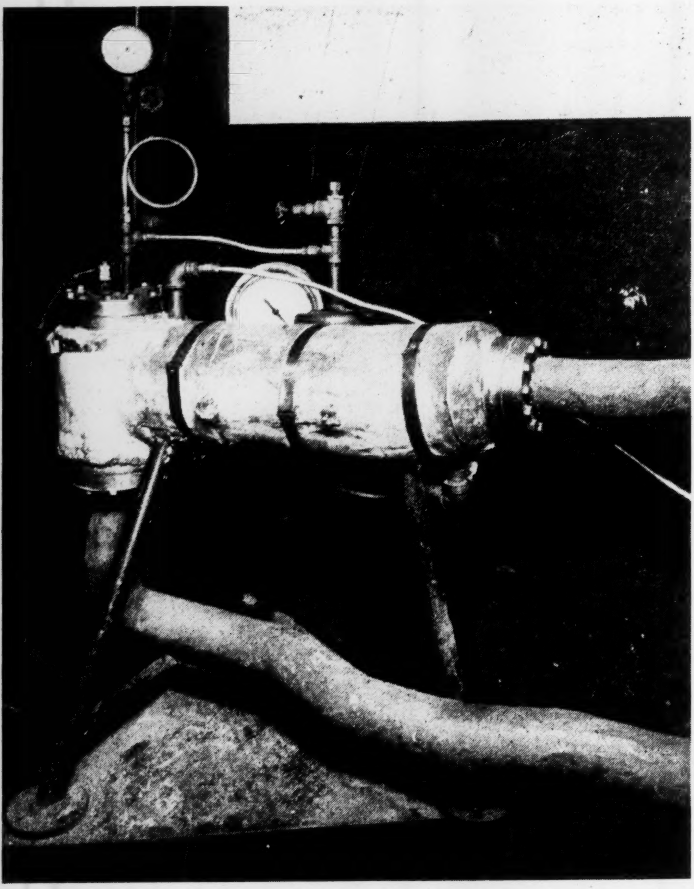

FIG. 11.—Cell in Use on a Study of Boiler Waters.

In Fig. 7 is shown a schematic installation on a drum sampling or boiler blowdown line from a 1400-psi. boiler. In this case a fixed resistance is installed to drop the pressure to a point slightly above that desired in the cell. The final control of the pressure in the cell is obtained through the use of a pressure-regulating valve in the cooling water line, set to maintain the desired pressure in the cell chamber. In our studies to date this pressure has been maintained at 2 or 3-psi. gage.

In Fig. 8 is shown the multiple orifice assembly used for the major pressure reduction. Experience to date indicates that with the present size and number of orifices, cleaning is advisable at about three-week intervals in continuous service.

Figure 9 shows the disassembled multiple orifice pressure reducer used in the studies to date.

Figure 10 shows schematically the apparatus as installed on a high-pressure heater for study.

Figure 11 shows the apparatus as installed to study its performance on a boiler-water sampling line from the drum of a 1400-psi. boiler. Just behind the condensing, or in this use cooling, cylinder can be seen the top of the cooling water regulating valve, the diaphragm of which is connected to the gage line on the cell chamber. The lead wires to the recorder can be seen at the left.

Figure 12 shows the recorder and its record as operating when attached to the apparatus of Fig. 11.

In each of the foregoing cases in which boiler waters or condensates were studied, the seal on the cell-chamber dis-

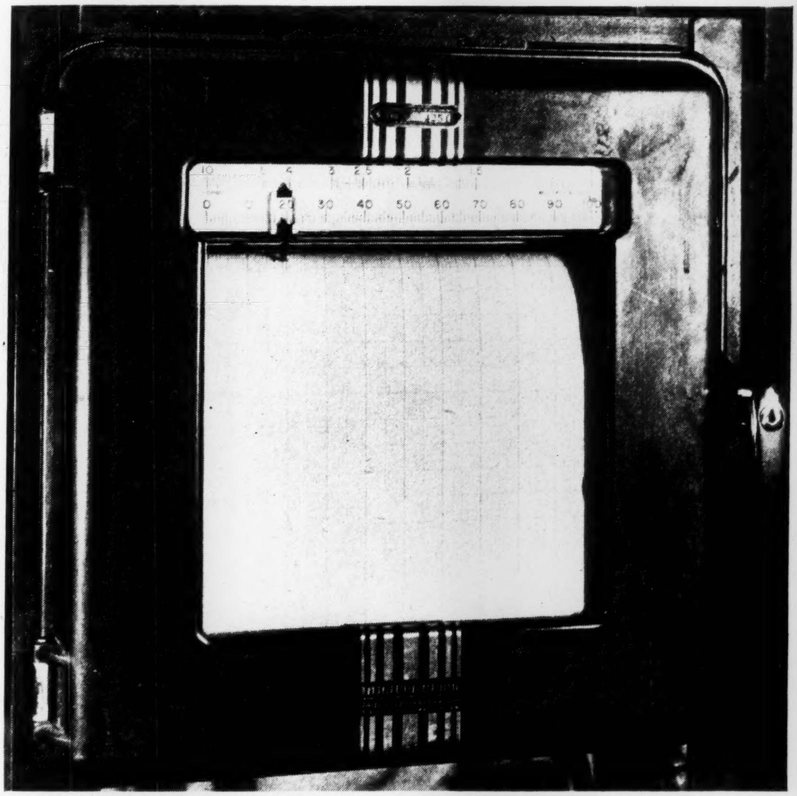

FIG. 12.—Recorder Showing Type of Record Produced.

charge was designed in accordance with the desired cell-chamber pressure.

As indicated in this description of a new type of conductivity apparatus, data have been collected over a period of about 2 yr. However, when the studies started, it was planned to investigate thoroughly the performance on steams made from waters that were 90 per cent raw and only 10 per cent condensate. These waters had no external treatment other than initial pH control and deaeration. The urgency of other questions indicated the advisability of transferring the equipment to condensates and boiler waters, and it is only recently that one of the cells has been reinstalled for studies of steam. Another cell is in use in connection with a study of silica control. In view of the fact that only an incomplete report of performance could be offered at this time, it has been decided to postpone a complete report on performance to a future meeting. However, some data are presented in the following paragraphs to indicate the trends.

TABLE II.—COMPARISON OF BOILER WATER ANALYSIS, TOTAL DISSOLVED SOLID CONCENTRATION AND CONDUCTIVITY RECORDER READINGS.

Date	Time	Sample	Boiler Water Analysis, ppm.				Dissolved Solids, ppm.		Conductivity Recorder Scale Reading
			$NaOH$	Na_3PO_4	$NaCl$	Na_2SO_4	By Addition	By Evaporation	
3-27-41	9:32	No. 1	25	25	72	215	337	...	435
	10:06	No. 2	29	25	90	220	364	...	440
	10:34	No. 3	28	25	94	195	342	...	438
	11:02	No. 4	23	25	97	185	330	...	430
	11:28	No. 5	21	25	98	180	324	...	420
	11:59	No. 6	19	20	100	165	304	349	400
	12:33	No. 7	19	20	96	145	280	...	385
	12:52	No. 8	16	10	93	130	249	283	357
	1:34	No. 9	14	10	86	105	215	...	330
	2:08	No. 10	12	10	79	95	196	...	310
	2:31	No. 11	11	10	76	95	192	209	300
	3:03	No. 12	10	10	72	105	197	...	305
	3:30	No. 13	10	10	61	110	191	...	305
	3:57	No. 14	10	10	62	125	207	...	305
3-28-41	9:37	No. 15	22	10	78	235	345	404	440
	10:08	No. 16	30	20	92	250	392	451	470
	10:48	No. 17	30	25	108	265	428	...	485
	11:44	No. 19	28	25	120	295	468	538	510
	12:10	No. 20	26	25	109	285	445	514	500
	12:33	No. 21	25	20	105	275	425	...	488
	1:34	No. 22	21	20	88	265	394	...	458
	2:10	No. 23	19	20	85	240	364	419	450

In Table II is shown a comparison of the total dissolved solid concentration as determined by analysis of samples taken at the time the scale reading of the conductivity recorder was noted. The analysis is expressed as the sodium salts and added to obtain the total concentration. It can be seen that, although there is general agreement between the proportions of the several components and the total, there are minor variations in composition. The amount determined by evaporation is consistently higher than that determined by addition of the components. The agreement of the total with the scale reading of the recorder is evident in spite of the chronological arrangement of the table.

TABLE III.—DATA OF TABLE II ARRANGED IN ASCENDING ORDER OF TOTAL DISSOLVED SOLIDS CONTENT.

Sample	Total Dissolved Solids in Boiler Water by Addition, ppm.	Conductivity Recorder Scale Reading
No. 13	191	305
No. 11	192	300
No. 10	196	310
No. 12	197	305
No. 14	207	305
No. 9	215	330
No. 8	249	357
No. 7	280	385
No. 6	304	400
No. 5	324	420
No. 4	330	430
No. 1	337	435
No. 3	342	438
No. 15	345	440
No. 2	364	440
No. 23	364	450
No. 16	392	470
No. 22	394	458
No. 21	425	488
No. 17	428	485
No. 20	445	500
No. 19	468	510

In Table III is shown the data of Table II rearranged in ascending order of the total dissolved solids. In this table the agreement of the recorder reading is more strikingly shown.

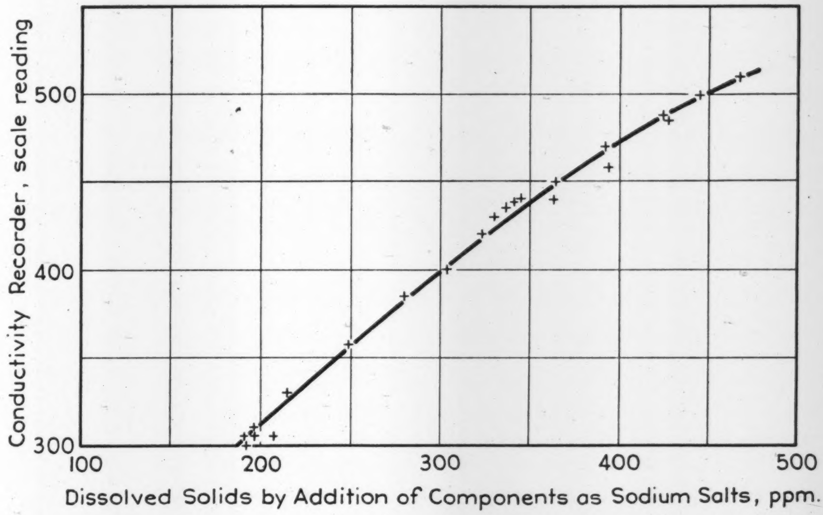

FIG. 13.—Recorder Reading *versus* Total Dissolved Solids.

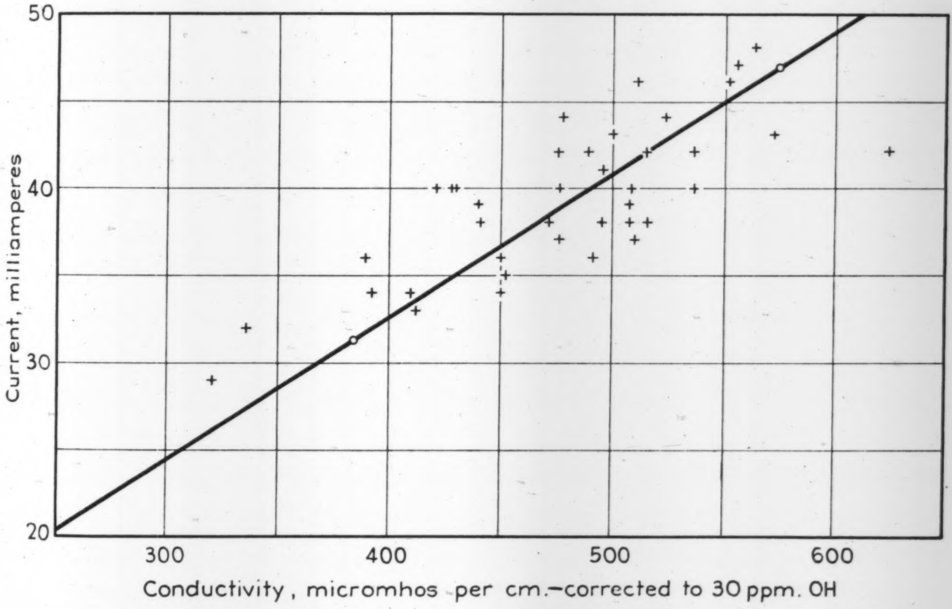

FIG. 14.—Current Through Cell *versus* Conductivity Corrected to 30 ppm. of OH.

In Fig. 13 the data of Table II are plotted.

We can conclude from these data: first, that the apparatus will produce a reading on boiler waters that can be plotted as a smooth curve against the concentration as determined chemically within the range shown; and second, that because no correction was made for hydroxyl ion, the correction for this factor becomes negligible even at 220 F. with the hydroxyl shown.

For those who might be interested in substituting a current measurement for the more expensive resistance measuring device, we have included Fig. 14. This figure shows the linear character of the relation between the current flowing and the conductivity corrected for OH ion.

TABLE IV.—FLOW OF COOLING WATER AND CONDENSATE.

Cooling water:	
Flow	17.5 lb. per min.
Temperature in	78 F.
Temperature out	184 F.
Heat absorbed	111,300 Btu. per hr.
Condensate:	
Steam condensed	137 lb. per hr.
Velocity through electrode cup	0.45 in. per sec.
Velocity through standpipe	11½ in. per. sec.

TABLE V.—LOG OF RESISTANCE AND CALORIMETER TEMPERATURE DURING PRIMING.

Time[a]	Resistance, ohms			Calorimeter Temperature, deg. Fahr.
	High-Pressure Cell	Glass Cell Before High-Pressure Cell	Glass Cell After High-Pressure Cell	
10:30 a.m.	4700	15 000	41 300	315
11:00 a.m.	3620	14 700	40 300	315
11:40 a.m.	3650	14 700	41 800	315
1:42 p.m.	4170	16 400	50 000	315
2:40 p.m.	4090	16 400	46 000	315
3:02 p.m.	2900	16 600	43 500	260
3:15 p.m.[b]	2700	13 500	22 600	252
4:00 p.m.	1600	14 500	20 800	323
4:30 p.m.	2300	15 300	26 300	322

[a] There is some question as to the setting of the chart on the calorimeter temperature recorder with respect to time.

[b] It was noted at 3:15 p.m. that the cell resistance was falling too fast for readings to be taken.

In Table IV are shown the conditions of operation of the cell during one test. With 17.5 lb. per min. flow of cooling water, condensate was formed at the rate of 137 lb. per hr., or sufficient to produce a velocity of about ½ in. per sec. in the electrode cup and almost 1 ft. per sec. in the standpipe.

In Table V is shown, as a matter of interest, a portion of the log of one test during which the boiler producing the steam primed heavily.

A glass cell, with its own condenser, was installed before the high-pressure cell, and another glass cell was installed after the high-pressure cell. Readings of each of the three cells were taken at intervals. The calorimeter temperature recorder chart was later examined, and the corresponding temperatures were noted. The rapid fall of all three resistances at the initiation of the priming period kept step with the calorimeter. Recovery was not complete at the end of the test period in any of the cells.

TABLE VI.—DATA FROM PRELIMINARY TEST.

CONDITION OF STEAM		
11:00 a.m.	CO_2 NH_3	undetermined 0.10 ppm.
2:45 p.m.	CO_2 NH_3	24 ppm. 0.10 ppm.
CONDITION OF CONDENSATE DISCHARGE		
11:40 a.m.	CO_2 NH_3	less than 1 ppm. 0.0 ppm.
	vent valve one-eighth turn open	
1:45 p.m.	CO_2 NH_3 pH	less than 1 ppm. 0.0 ppm. 6.8
	vent valve one-eighth turn open	

In Table VI are shown some data on the effect of the apparatus on gaseous contaminants. Apparently the steam contained about 24 ppm. of carbon dioxide and 0.10 ppm. of ammonia. The condensate contained less than 1 ppm. of carbon dioxide and 0.0 ppm. of ammonia. The analysis for CO_2 was made by the method developed for the National District Heating Association and now is being considered for adoption by this Society. The significance of the phrase "less than 1 ppm." would normally be considered 0.0, except for the development status of the analytical equipment.

The artificial contamination of the steam supply is being considered to determine the influence of the amount of contaminants on the required venting. It will be noted that the needle valve controlling the venting during these observations was opened only one-eighth of a turn.

Acknowledgment:

The author wishes to express his deep appreciation to members of the Research Bureau and of the Technical Service Dept. for their assistance, and especially to A. A. Markson, Thomas Ravese and H. W. Betzler for their invaluable aid in the development and application of the apparatus, and to Thomas J. Finnegan and A. S. Bostwick for carrying on the studies of the performance of this equipment.

[See Joint Discussion, p. 1337.—ED.]

CONDUCTIVITY CELLS AND ELECTRICAL MEASURING INSTRUMENTS

By C. E. Kaufman[1]

The science of conductivity measurement, solidly founded on the researches of Kohlrausch (**32**)[2], and extended by the work of numerous subsequent investigators, has for years interested those concerned with the quantity of electrolytes dissolved in industrial waters. The chief advantages of this method of analysis are the rapidity and comparative accuracy with which measurements can be made and the opportunity for easily following and automatically recording water conditions continuously over considerable intervals of time.

Though the measurement of conductivity is a science, application of the method to particular problems in industrial water treatment is still somewhat of an art, requiring selection of suitable instruments and methods and the proper interpretation of results (**18, 20, 21, 27, 30, 44, 45, 55, 70**). It is the purpose of this paper to point out the character of some of the cells and instruments commercially available for electrical conductivity measurements, their limitations, and some of their uses. First, however, the question of precision will be briefly considered.

Relating Measurement to Operating Problems

What does the engineer desire from a measurement of electrical conductivity on a particular sample of water? Rarely will he be concerned with the conductivity *per se*; usually he will be seeking an answer to a specific question: When must a condenser be taken off the line to repair leaks? Can blowdown from a boiler be decreased or must it be increased? Are deposits going to accumulate in superheater tubes or on turbine blades?

The answer to any such question can scarcely be supplied merely by measuring the resistance between two electrodes immersed in a sample of the water in question. Beyond the factor of *measurement* lies the factor of *judgment* based upon experience. For example, little correlation has yet been found between the conductivity of condensed steam and the rate of accumulation of turbine deposits. Much attention has been paid to the correction for, or elimination of, dissolved gases which contribute to the conductivity of steam condensate, as discussed in companion papers in the present symposium[3, 4, 5, 6] and also in the literature (**29, 46, 47, 57, 59, 74**). Greatest emphasis at the present time is being given to the problem of eliminating automatically from the sample all gaseous impurities, whose conducting effect often outweighs that of the dissolved solids. Powell

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[1] Research Engineer, Hall Laboratories, Inc., Pittsburgh, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the bibliography appended to this symposium, see p. 1338.

[3] S. F. Whirl and W. A. Lower, "Experimental Methods of Determining Conductivity Corrections for Dissolved Gases in Steam Condensate," see p. 1277.

[4] D. S. McKinney, "Calculation of Corrections to Conductivity Measurements for Dissolved Gases," see p. 1285.

[5] P. B. Place, "The Degasification of Steam Samples for Conductivity Tests," see p. 1302.

[6] A. R. Mumford, "A New Type of Conductivity Apparatus for Use with Boiler Waters and Steam Samples," see p. 1314.

(47), Gurney (20), Straub (63), Mumford,[6] and co-workers have attacked this problem from various angles of approach. Even though the contribution of dissolved gases to the conductivity may be eliminated, it would be a rash chemist or engineer who, looking at a measurement made in a plant the history of which he did not know, would dare to guess what degree of trouble had been encountered from carry-over of dissolved solids.

If judgment based upon experience is an essential factor in the interpretation of a measurement of conductivity, the question at once arises: How precise need the measurement be in any particular case to supply a sound basis for the exercise of this judgment? In so far as research is concerned, precision may lead to new information of great value; but, just as pipe can be fitted quite adequately without using Johansson gages, a Jones bridge and a cell thermostatically regulated to plus or minus 0.001 C. are scarcely necessary to control blowdown from a boiler.

It is perhaps heresy to argue that too great precision in measurement may be an actual handicap; and yet, in practical control, unimportant minor variations observed with equipment of high sensitivity may occasion long arguments and investigations which represent a real waste of technical effort. In applying the conductivity method, it may therefore be well to select equipment with an eye to the job to be done and to restrain the natural impulse toward progressive refinement of every measurement; the latter may lead to worship of precision for it's own sake rather than to emphasis upon the meaning behind the measurement.

Since every measurement of electrical conductivity is fundamentally a measurement of resistance, it may be useful to consider separately the cell that provides the resistance and the instrument that measures it.

THE CONDUCTIVITY CELL

In theory, the conductivity cell is a mechanical device for isolating a column of the solution whose resistance is to be measured, so that the cross-sectional area and length of this column shall be constant and there shall be no contact resistance at the electrodes. In practice, the conductivity cell is simply a containing vessel in which two electrodes are fixed. The actual cell is related to the theoretical cell by calibration with solutions whose conductivities are already known with precision (28), yielding the cell constant by which all resistances measured in the cell must be divided to obtain values for resistivity.[7]

For results of the highest precision, it is probably advisable to calibrate the cell with a KCl solution whose conductivity is near that of the unknown solutions to be measured. It may also be of advantage to calibrate at or near the temperature at which measurements are to be taken, since expansion or contraction of the electrodes and cell body may affect results. In the event that an accurately calibrated cell is available, other cells can of course be standardized against it.

If the resistance of the unknown solution is to be measured properly, there must be no additional resistance of a "loose contact" between the solution and electrodes caused by deposits of grease, dirt, or scale screening the metal surface, or by polarization with a gas film resulting from the passage of electric current through the solution. On the other hand, the resistance must not be decreased by the introduction into the solution of ions derived from sampling connections, the atmosphere, or from the materials of the cell itself, or by electrical leakage between the electrodes or leads. Finally, it is necessary to remember that the resistance of the solution changes with temperature, and hence that no conductivity value has a precise significance unless the temperature of the solution is known.

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[7] Some confusion may be avoided if it is remembered that the actual measurement is resistance in ohms, that by the use of the cell constant this is converted into resistivity in ohm square centimeter per centimeter, and that the reciprocal of the resistivity is the conductivity, the units for which are centimeter per ohm square centimeter, or mho centimeter per square centimeter. Since the use of the mho unit usually leads to the expression of results in awkwardly small numbers, the term micromho, 1×10^{-6} mho, is frequently employed. Reporting of results in terms of conductivity is to be preferred to their expression in resistivity units, primarily because conductivity values increase directly with dissolved solids content whereas the corresponding values of resistivity vary inversely with the content of dissolved material.

It might be pointed out that "conductivity" and "specific conductance" are synonymous, but that the former term is preferred; also, a mho or a micromho is a unit of conductance rather than of conductivity, the latter being expressed in terms of mho centimeter per square centimeter, which reduces to mho per centimeter.

The following factors may be considered as establishing the design of a conductivity cell or electrode system:

1. Shape of cell and size and spacing of electrodes.
2. Material of construction of electrodes.
3. Material of construction of cell body.
4. Accessories.

Shape of Cell and Size and Spacing of Electrodes:

The building of conductivity cells has offered a fertile field for ingenious designers, and their resultant products sometimes bear only a slight family resemblance to one another. For industrial purposes, conventional laboratory and research cells are ill-suited because of fragility. More generally useful are dip cells, ordinarily comprising a protective sheath surrounding the two electrodes. Such cells can easily be adapted to continuous flow devices by enclosing in a chamber such as that in Fig. 1. For permanent industrial locations and continuous service, cell assemblies are available for inserting in pipe lines or tanks. Since electrodes should be periodically cleaned and recalibrated, some designs provide for removal of the electrodes proper without disturbance to continued operation in the line or tank in which electrolytic strength is being measured.

In all cells, the size, shape, and spacing of the electrode elements proper and the shape of the containing body or vessel determine the cell constant. In general, with solutions containing an appreciable amount of electrolytes, electrodes can be relatively small and far apart, while with solutions of low concentration, larger and closer-set electrodes can be used. Some cells have been designed with a variable distance between electrodes.

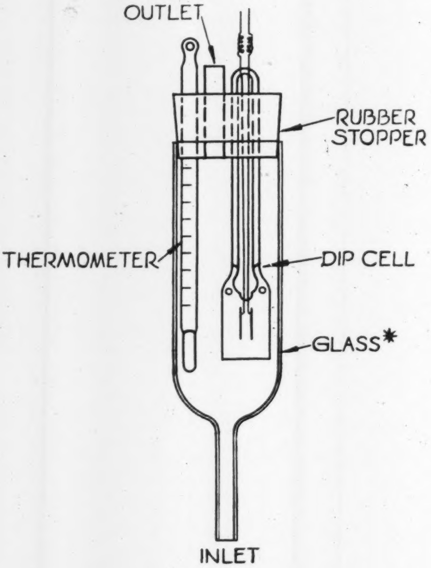

FIG. 1.—Simple Flow Cell.

* Pyrex or other sparingly soluble glass preferred.

While cells ordinarily operate at fairly low temperatures, modified designs have been proposed for use at the elevated temperatures and pressures prevailing in boilers.[6, 8]

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[8] Henry C. Parker, U. S. Patent 1,898,209 (1933).

A most important point in the design of flow cells is to make the electrode chamber small in order to avoid lags in reading and the "leveling" effect of a large reservoir of water, which may be made up of portions of widely different conductivity.

Material of Construction of Electrodes:

The ideal material for electrodes to be used in conductivity measurements would have the following properties:

1. Good conductance of electricity.
2. Resistance to corrosion by the medium being measured. (Such corrosion might alter the cell constant as well as add conducting material to the solution.)
3. Resistance to erosion (same reasons as for corrosion resistance).
4. Sufficient strength so that the shape of, and spacing between, electrodes cannot inadvertently be altered.
5. A surface structure that tends to eliminate polarization error and does not promote adsorption.

The materials most commonly used for electrodes are platinized platinum, smooth platinum, gold, stainless steel, and monel metal. For general use under a variety of conditions, platinized platinum is probably of greatest value because it minimizes polarization error. Smooth platinum, gold, stainless steel, and monel metal have been employed, particularly where the conductivity is very low, as in condensed steam or condensate, since the polarization error usually decreases with decreasing measured conductivity; even in solutions of higher concentration results of adequate accuracy can be obtained in many cases. It is possible that many other metals or alloys can be used, especially in process work where special problems of corrosion confront the electrode user.

Material of Construction of Cell Body:

While supporting the electrodes rigidly, suitably insulating them from each other, and in some cases providing protection, the cell body must meet the same requirements of strength and resistance to corrosion and erosion as the electrodes themselves. Different kinds of glass, plastics, hard rubber, and metals have been used in constructing bodies and casings. The electrodes may, for example, be mounted in glass or bakelite with a stainless steel or nickel protecting sheath. Cells constructed from hard rubber or plastic material have been described by Walker (73) and Collins (7).

In all cases, the body should be so constructed that the electrode surfaces can be easily inspected and cleaned.

For many practical purposes, an apparatus no more elaborate than a pair of stainless steel or monel metal rods in a holder of rubber or plastic will prove useful. Ulmer (71) has described one simple arrangement of this type.

In investigating the utility of plastic cell bodies, attention should be devoted to their resistance to hot solutions and to the possibility of adsorption of acids, alkalies, or other materials which might affect subsequent readings.

Accessories:

Although a thermometer mounted integrally with the cell or nearby is convenient when a temperature correction is necessary, most industrial cells do not have attached thermometers. Figure 1 illustrates how a thermometer can easily be mounted in a chamber flow cell.

Lead wires should be well insulated, preferably rubber-covered, and should present no opening at the point of attachment to the cell body where moisture or other substance could penetrate and subsequently cause shorting.

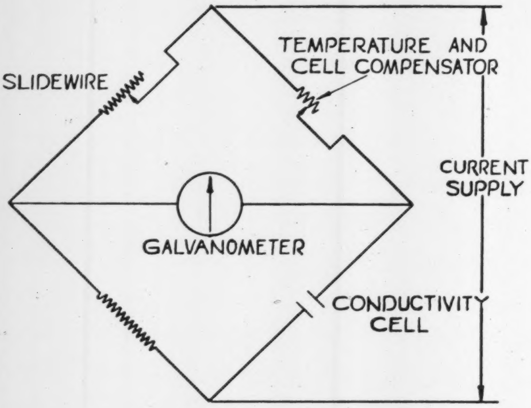

Fig. 2.—Wheatstone Bridge Circuit.

The Measuring Instrument

To measure the resistance between the electrodes in the cell, current must flow through the system. Since direct current would unavoidably tend to polarize the electrodes, alternating current is employed at frequencies ranging from 1000 cycles for research investigations down to 60 cycles for most industrial work, and in some cases 25 cycles where this is the only available supply.

Of the two principal classes of measuring devices, the Wheatstone bridge and the deflection meter, the former is inherently more accurate, and precise measurements can be made with the passage of much smaller currents through the test solution. The latter, by virtue of its off-balance reading, is limited in accuracy by meter design and voltage fluctuations. A simple Wheatstone bridge circuit is illustrated in Fig. 2 and a deflection hookup in Fig. 3. Wheatstone bridges measure the actual total resistance of the cell; deflection instruments read the current passing through the cell. The resistance calculated from the voltage and current is not necessarily the resistance of the cell but may include other circuit resistances. The following list classifies the general kinds of conductivity measuring equipment commercially available[9] or easily constructed:

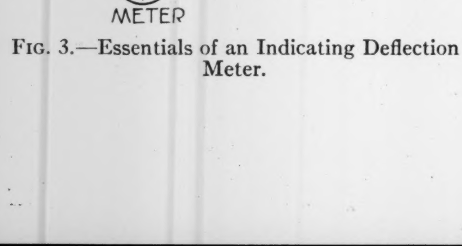

Fig. 3.—Essentials of an Indicating Deflection Meter.

A. Wheatstone bridges (balance instruments):
    1. Manually adjusted.
    2. Recording.
    3. Controlling or warning.

B. Current meters (deflection instruments):
    1. Indicating types utilizing voltmeters and ammeters.
    2. Recording.
    3. Controlling or warning.

C. Glow lamp arrangements.

Wheatstone Bridges:

The reference instrument for conductivity and resistivity measurements is the

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[9] The reader is referred to the current literature of instrument and cell manufacturers, among whom are the following: Allis-Chalmers Manufacturing Co., Dearborn Chemical Co., Esterline-Angus Co., Inc., Industrial Instruments, Inc., Leeds & Northrup Co., National Aluminate Corp.

Wheatstone bridge, a fundamental circuit for determining unknown resistances. In the best research instruments, resistances are finely calibrated and the imposed alternating current is of high frequency (of the order of 1000 cycles per second) in order to cut down polarization error. In at least one industrial bridge, a vacuum tube oscillator has been adopted for use in supplying 1000 cycle measuring current from a 60 or 25-cycle-source. When vacuum tube amplification is used in special equipment, extensive shielding has been recommended to reduce capacitance errors; but this procedure does not appear to be necessary in less exacting industrial measurements. In research bridges grounding may also be resorted to for minimizing capacitance effects. In many industrial bridges insulating transformers are provided to avoid the necessity of insulating the test solution from ground when the power line is grounded. With all refinements of design, the Wheatstone bridge is more complex and undoubtedly more expensive than is necessary for ordinary testing. For industrial purposes there is available a considerable range of less precise but adequate equipment.

Relatively inexpensive units, manually operated, are available for specific uses such as testing distilled water and condensate, determining dissolved solids in more concentrated solutions, and checking concentrations of the active substance in process waters. Certain of these instruments have been built with cathode-ray tubes as the null-point indicator instead of the more usual galvanometer. Since such apparatus embodies a bridge circuit, usually operated on low voltage from a transformer, voltage fluctuations are of little importance.

Temperature variation of the electrolyte to be measured is of considerable importance, due to the fact that conductivity increases by about 2 per cent for a rise in temperature of 1 C. over the limited temperature range in which most measurements are made. A number of industrial Wheatstone bridges are provided with temperature compensators which cover ranges ordinarily encountered. The general principle on which bridge compensators operate can be seen from Fig. 2. For example, if the solution in the cell is at a temperature higher than the standard value of 25 C. (77 F.), the conductance of the arm in which the cell is located will be increased (the resistance decreased). The compensator is turned till it is equivalent to the measured temperature of the electrolyte. This serves to decrease the resistance of the arm in which the compensator is located so that the ratio of the resistances at the temperature of measurement is approximately the same as the ratio at the standard temperature. This type of compensation, while independent of the concentration of electrolyte in the solution to be measured, involves the assumption, probably justifiable for ordinary plant work, that all electrolytes have identical temperature coefficients. A good bridge compensator will also be so designed that correction is made on the basis of the actual average change of resistance with temperature rather than by a simpler but erroneous linear function.

Bridges often are equipped with cell compensators which permit direct reading of the instrument when the electrode systems employed vary in cell constant over a limited range. Another type of manually operated compensator, designed to correct for changes of dissolved CO_2 content in condensate, is mentioned by Hecht and McKinney (27). Compensation for other dissolved gases or for combinations of gases could be devised.

Instruments can be obtained calibrated in terms of parts per million or grains per gallon dissolved solids (sometimes expressed as concentration of a particular electrolyte), conductivity, resistivity, measured resistance, or with double-reading scales showing both conductivity and resistivity. In general, where an all-purpose instrument is desired which puts no preconceived interpretation on the results, readings should be in terms of conductivity, resistivity, or measured resistance rather than in a specialized unit such as parts per million of NaCl, which involves the use of an arbitrary conversion factor.

Portability is often a factor in the selection of a general utility instrument. Equipment is sometimes manufactured with integral carrying case, while special cases may be provided for other apparatus.

For one portable conductivity bridge now on the market, an accuracy of adjustment for the conductivity scale of 0.3 per cent of its range is claimed. The accuracy of adjustment of the cell constant compensator is estimated to be about 2 per cent for the value of about 0.1, the constant of the cell ordinarily employed, while the temperature compensator is accurate to 1 C., corresponding to about 2 per cent. The over-all conductivity range is from 0.012 to 0.0000001 mho square centimeter per centimeter (resistivity from about 80 to 10,000,000 ohm square centimeter per centimeter). Such an instrument is considered adequate for the most important and exacting carry-over determinations.

Recording Instruments of the Bridge Type:

Where a continuous and permanent record of concentration of process water, purity of condensed steam, or conditions in some other industrial water is desired, the Wheatstone bridge circuit can be adapted for recording of conductivity, resistivity, resistance, or some function thereof. In such installations synchronous motors are usually employed to turn recording charts. Balance of the bridge is ordinarily maintained by means of an automatically functioning circuit actuated by deflection of the bridge galvanometer. Temperature correction on present models is ordinarily manual, which may be a drawback when conditions cannot be readily controlled. However, Powell and co-workers (47) have described a two-point recording conductivity bridge which is automatically compensated for temperature by means of resistance thermometers installed in the cells. The thermometers are connected to temperature recorders which actuate correcting slidewires in the conductivity bridge circuit.

Where recording instruments are required to show the ratio between the actual strength of a solution and the desired concentration, two cell assemblies are used. A reference cell contains solution at standard concentration, while the other electrodes are placed in contact with the solution being measured. This arrangement provides the best temperature compensation when properly applied and also reduces polarization errors.

Multipoint recorders are an especially interesting development, since their employment enables an operator to follow conditions simultaneously throughout a system. Recorders can be important aids in routine control and are of the greatest advantage in making surveys of operating conditions. However, due to their cost, such instruments are not yet commonly in use.

Controlling Instruments of the Bridge Type:

Recording Wheatstone bridge installations can be further adapted to control the addition of treatment materials or to actuate warning signals when concentrations of measured solutions vary from a predetermined level. Where automatic or semi-automatic control of processes is desirable, such instruments should find wide application. Manually adjusted bridges have also been adapted to operate warning lights (72).

Current Meters:

The simplest means of finding the approximate conductance or resistance of a solution involves nothing more than measuring the current when an open circuit with a source of imposed voltage is closed by inserting two electrodes into a solution to be measured. Intrinsically such a system, illustrated in Fig. 3, may not be as accurate as a bridge circuit, but its low cost, simplicity, and adaptability make its use attractive where feasible. Temperature compensation within the instrument is more involved than in a Wheatstone bridge circuit, since the correction depends not only on the temperature but also on the concentration. Temperature correction, of course, can be made by multiplying the measured reading by a suitable factor, related to the temperature of the sample.

Since the simplest deflection instruments are based on a series circuit, fluctuations in the imposed voltage may seriously influence the results. Therefore, it is advisable to select an instrument which has some means of adjusting or compensating for voltage changes. Suitable variable resistances or constant-voltage supply transformers can be utilized for this service. Deflection instruments can be calibrated in any desired fashion: conductivity, resistivity, resistance, current, or parts per million or grains per gallon of a particular constituent, although fundamentally one of the first four is preferable.

Recording Instruments of the Deflection Type:

Recording ammeters (69) and ohmmeters have been adapted for obtaining continuous resistance readings of electrolytes varying in strength from condensed steam to boiler water, although cells are commonly changed for extreme ranges of concentration. In certain electrodynamometer circuits, it is claimed that line voltage fluctuations are not important since the design is in a sense a "bridge" system. The accuracy of instruments of this type is reported to be of the order of 1 per cent of the full scale deflection. Spring clocks and synchronous motors are commonly used to drive the recording chart.

Although not now believed to be on the market, another type of arrangement which might be set up involves the use of an indicating galvanometer to show current or voltage change in conjunction with a sensitive photoelectric recorder which has already been developed. In such a system, deflection of the galvanometer mirror sends light through an optical system to phototubes which actuate an electron-tube circuit, which in turn controls the recorder.

A possible means by which fairly continuous readings could be recorded when only an indicating deflection instrument is available involves the use of a suitably controlled camera mechanism to photograph the position of the pointer on the scale at regular intervals.

Controlling or Warning Instruments of the Deflection Type:

In addition to indicating changes of conductivity or related properties, instruments have been devised which will also operate controls and make warning signals. For example, it is possible with indicators of this type to open valves, ring bells, or flash lights. In the future, there will be, no doubt, a considerable increase in this type of installation.

Glow Lamp Arrangements:

It is possible to put a lamp or lamps in series with two electrodes and a source of current in order to obtain a rough measure of the conductivity of an electrolyte. The electrodes can be so arranged, for example, that a dim glow in a lamp indicates the building up of a known concentration. In constructing a homemade device of this type, care should be exercised in order to avoid dangerous shocks or the possibility of electrolysis. A similar type of instrument for blowdown control in locomotives employs two sets of electrodes, one shorter than the other, mounted at the top of the boiler above the normal water level. When foaming or priming starts, the liquid thrown up first closes the circuit of the electrodes extending farthest down, causing a light to glow and a blowdown valve to open. When liquid is thrown even higher, another warning light goes on.

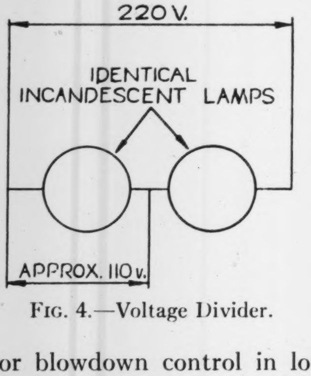

FIG. 4.—Voltage Divider.

Special Apparatus:

Occasionally, in field tests, only 220-v. service is available, while measuring instruments are designed for use at 110 v. In such a case, the simple divider shown in Fig. 4 can be used to supply approximately the desired potential.

Where only direct current is available, it is possible to produce the requisite alternating current by means of a vacuum tube oscillator or by use of a suitable motor-generator set.

FITTING THE EQUIPMENT TO THE JOB

Recognizing the likelihood that there will be at least as many opinions concerning the type of equipment needed for a particular purpose as there are different units on the market, the author ventures some suggestions for several typical applications:

Determination of Steam Purity:

An acceptance test on a new high-capacity boiler in a central station may require elaborate precautions to eliminate dissolved gases from the condensed steam, together with precision equipment of the bridge type to measure the conductivity due to dissolved solids, in order that ability to meet a stringent guarantee on steam quality may be proved. A suitable degassing and automatic recording unit may even be permanently installed to give a continuous record of conditions during operation. By employing suitable nozzles, sampling lines, and flow cells with a multipoint recorder, steam going to the superheater, steam leaving the superheater, and condensate leaving the condenser, as well as raw water and feedwater may be checked throughout the operating life of the unit.

Instrumentation of this sort is obviously valuable from the research as well as the operating viewpoint. Indeed, its expense may be justifiable only where the installation is supervised by a chemist or engineer not only qualified by training but driven by an inquiring mind. Such a man will recognize, not only the utility of the conductivity method in measuring dissolved salts, but also the uncertainties in the subsequent interpretation into terms of operating difficulties.

New industrial power plants containing a small number of large boiler units fall in the same category as their contemporary central stations. In many industrial plants, however, particularly where a considerable number of boilers are in one or more locations, portable equipment is more practical. The emphasis again should fall on the knowledge and experience of the man behind the measurement; even though a good man deserves good tools, he can, if necessary, correctly analyze a situation with equipment assembled from any electrical or radio supply store, while a man inadequately prepared for the job may easily misinterpret data obtained with the finest equipment.

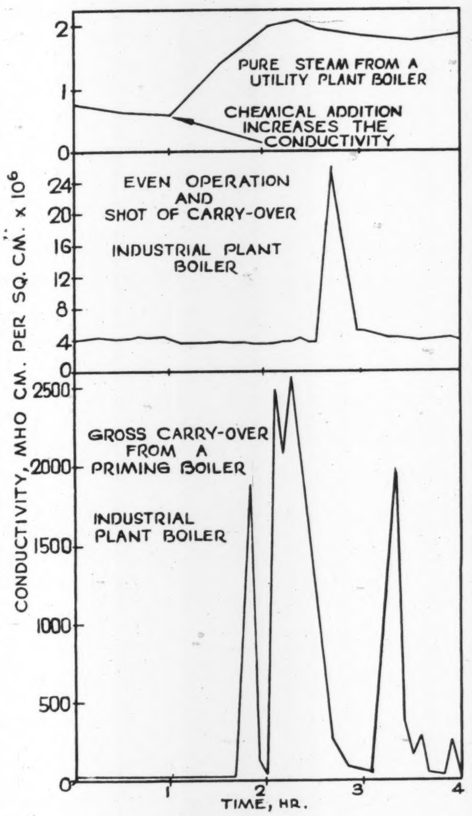

FIG. 5.—Conductivity Tests on Condensed Degassed Steam from Three Different Boiler Plants.

Portable recording conductivity meters relieve the tedium of repeated measurement by manual adjustment, but the instrument cannot yet substitute completely for the man; all it can do is to record what it measures in the conductivity cell; the man must decide whether the increase in conductivity at 4 a.m. was due to increase in temperature of the sample because of decreased flow of cooling water, to increase in the content of dissolved gases in the sample because of variation in softener operation or efficiency of a degassing unit, or to actual carry-over of dissolved salts from the boiler.

Figure 5 illustrates conditions which may be met in the application of the conductivity method. "Steam" may run the gamut from a nearly perfect product to one with most of the characteristics of boiler water.

Testing for Condenser Leakage and Condensate Purity:

The same apparatus used for steam tests may, of course, be applied for this purpose. For routine checking, the use of indicating deflection meters may be convenient.

Estimation of Dissolved Solids Content of Boiler Water and Control of Blowdown:

In the estimation of dissolved solids of boiler water, even when samples are neutralized and considerably diluted, electrolytic strengths are high and polarization error becomes more important. There is also usually a certain degree of uncertainty in correlation of conductivity with dissolved solids, especially since considerable amounts of nonionized material such as tannin, lignin, or other organic material and perhaps silica may be present and yet be undetected by the conductivity method. Even though readings may consistently exhibit an absolute error, if results are consistent and reproducible, they are very likely to be adequate for plant control. In view of the inherent errors under these circumstances, it is doubtful if the use of precise equipment is justified. In many cases, simple indicating deflection instruments can be used to advantage.

Control of Plant Waters and Solutions:

Routine checks of raw water, feedwater, and process solutions may all be made with relatively simple indicating deflection equipment. Such measurements cannot always be related accurately to definite values of conductivity or resistivity, but they can serve as a continuing check on the performance of plant units and treatment and as an indicator of contamination.

Conductimetric Titrations:

Even the simplest conductivity equipment can be employed in running conductimetric titrations, for example, of alkalinity. Indicating deflection apparatus may be more convenient for this purpose than more accurate bridges which require manual balancing.

CONCLUSION

In selecting instruments and cells, the demands for accuracy and convenience must necessarily be satisfied as economically as possible. Of course, this does not necessarily mean purchasing the cheapest apparatus, but rather that which will best do the work and stand up in continuous service. Sometimes the purchase of accurate all-purpose equipment is justified by the opportunities offered for wide application. In other cases relatively simple apparatus will yield results adequate for particular uses.

While cells are already available in a variety of designs and materials, it is to be expected that there will be considerable development in the future of the newer plastics and alloys in forming rugged, corrosion-resisting flow cells for industrial usage.

Of the two principal types of instrument, the Wheatstone bridge and the deflection meter, the former is recognized as inherently the more stable and accurate and is to be recommended for precise measurement. Recorders and controllers embodying this principle will no doubt come into more prominent use on account of their convenience. Deflection instruments, especially those of the simple and inexpensive indicating type, can be widely applied in routine checking and control.

In brief, it has been the aim of the author to point out the general types of conductivity equipment available and to mention some of the factors to be considered in choosing apparatus for particular applications.

JOINT DISCUSSION

A New Type of Conductivity Apparatus for Use with Boiler Waters and Steam Samples

By A. R. Mumford

Conductivity Cells and Electrical Measuring Instruments

By C. E. Kaufman

Mr. R. T. Hanlon.[1]—A review of the results obtained using the high-temperature conductivity cell developed by Mr. Mumford may be helpful in indicating the applicability of this cell to evaluation of boiler water soluble solid concentration.

We paralleled the high-temperature cell with a cooling coil in conjunction with an ordinary liquid type conductivity cell and cooled the boiler water passing through this cell to 25 C. A comparison of simultaneous conductivity determinations using these two cells showed that the conductivity values obtained using the Mumford cell increased and decreased in proportion to those obtained with the sample at 25 C. In this manner the performance of the Mumford cell was correlated with the conductivity data as given in the International Critical Tables.

Mr. Kaufman has submitted an excellent paper outlining the various means by which conductivity may be measured. In connection with this paper, too much emphasis cannot be placed upon the calibration procedure followed in standardizing cells for conductivity measurement. The use of the conductivity method for determination of steam quality has progressed beyond the point where relative samples taken at scattered points, and not simultaneously, are considered adequate for accurate steam-quality measurement. It is now common practice to obtain simultaneous samples across a boiler drum in the manner prescribed by Baker (**3**)[2] or to make simultaneous conductivity determinations at various points in the steam cycle for comparative purposes. Hence, accurate cell standardization procedures which will yield reproducible results must be employed.

Relative to conversion of conductivity values to parts per million soluble solids, our experience indicates that such a practice should not be encouraged until it is possible to submit sound laboratory results upon which such conversion should be predicated.

Mr. A. R. Mumford[3] (*author's closure, by letter*).—The author is very appreciative of the kind of work Mr. Hanlon mentions as having been done in extending the information concerning the operation of the new cell. It was unfortunate that the pressure of other activities and demands for use of the equipment prevented the accumulation of all the data desired before the publication of the descriptive matter took place. It is to be hoped that extended performance data will be collected and made available to confirm the preliminary indications of satisfactory performance.

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[1] Chemist, Consolidated Edison Company of New York, Inc., New York, N. Y.

[2] The boldface number in parentheses refers to a paper appearing in the bibliography appended to this symposium, see p. 1338.

[3] Senior Research Associate, Consolidated Edison Company of New York, Inc., New York, N. Y.

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SUMMARY OF PROCEEDINGS OF THE WASHINGTON SPRING MEETING

SYMPOSIUM ON NEW METHODS FOR PARTICLE SIZE DETERMINATIONS IN THE SUBSIEVE RANGE

SYMPOSIUM ON COLOR—ITS SPECIFICATION AND USE IN EVALUATING THE APPEARANCE OF MATERIALS

The twelfth spring meeting of the Society was held in Washington, D. C. at The Mayflower, Tuesday, March 4, and Wednesday, March 5, 1941, in conjunction with the Group Meetings of the A.S.T.M. Committees. The spring meeting consisted of two formal technical symposiums—one on Particle Size, the other on Color.

Symposium on Particle Size:

The afternoon and evening sessions on Tuesday, March 4, were devoted to the Symposium on New Methods for Particle Size Determinations in the Subsieve Range. This symposium was developed under the auspices of the Section on Pigment-Type Materials of Technical Committee III on Particle Size and Shape, of Committee E-1 on Methods of Testing of the American Society for Testing Materials, C. E. Barnett (*Chairman*), The New Jersey Zinc Co. (of Pennsylvania).

The development of satisfactory, practical test methods for the determination of particle sizes must depend upon the research of individual investigators. In the past few years there has been much activity in this work and consideration to those procedures which appear to have possibilities as specification methods. To this end the following seven papers in this symposium cover the measurement of particles varying all the way from submicroscopic up to those which can be separated by screening:

"Methods for Determining Particle Size Distribution," by Herbert E. Schweyer, Graduate Student, Columbia University, and Lincoln T. Work, Director of Research and Development, Metal and Thermit Corp.

"Shape and Surface of Fine Powders by the Permeability Method," by P. C. Carman, University of Cape Town (South Africa).

"Surface Area of Portland Cement," by Paul S. Roller, Physical Chemist, Eastern Experiment Station, U. S. Bureau of Mines, and P. V. Roundy, Jr., Cooperative Fellow, American Instrument Co.

"The Determinations of Subsieve Particle Size Distributions by Sedimentation Methods," by S. W. Martin, Research Chemist, Titanium Division, National Lead Co.

"The Electron Microscope in the Determination of Particle Size Characteristics," by James Hillier, Research Laboratories, RCA Manufacturing Co.

"A New Method for Measuring the Surface Areas of Finely Divided Materials and for Determining the Size of Particles," by P. H. Emmett, Professor, Department of Chemical and Gas Engineering, The Johns Hopkins University.

"Specific Surface of Pigments by Adsorption from Solution," by Warren W. Ewing, Professor of Physical Chemistry, Lehigh University.

The presiding officers for the sessions were Lincoln T. Work and C. E. Barnett.

Symposium on Color:

An afternoon session on Wednesday, March 5, of the meeting comprised a Symposium on Color—Its Specification and Use in Evaluating the Appearance of Materials. This symposium was jointly sponsored by the Society and the Inter-Society Color Council. The Inter-Society Color Council is an organization of national societies having an interest in color. The aim of the council is to coordinate and to stimulate the work on color which is being done in the member bodies.

The joint committee in charge of the development of the technical program consisted of the following:

M. Rea Paul (*Chairman*), National Lead Co.
A. G. Ashcroft, Product Engineer, Alexander Smith and Sons Carpet Co.
W. E. Emley, Chief, Division of Fibrous and Organic Materials, National Bureau of Standards.
C. E. Foss, Interchemical Corp.
W. R. Fuller, Technical Director, Grand Rapids Varnish Corp.
H. A. Gardner, Director, Scientific Section, National Paint, Varnish and Lacquer Assn., Inc.
Deane B. Judd, Physicist, National Bureau of Standards.
Richard S. Hunter, Assistant Physicist, National Bureau of Standards.
Paul Rapp, Associate Chemist, Public Roads Administration, Federal Works Agency.
W. M. Scott, Chief, Cotton Chemical Finishing Division, U. S. Bureau of Agricultural Chemistry and Engineering.

The following six papers in this symposium cover practically all phases of color, and stress those aspects of primary interest to the specifier of appearance attributes of color:

"Introduction to Color," by Deane B. Judd, Physicist, National Bureau of Standards.

"Color Specifications of Transparent Materials," by Francis Scofield, Chemist, National Paint, Varnish and Lacquer Assn., Inc.

"Hiding Power and Opacity," by Roscoe H. Sawyer, Research Division Head, Krebs Pigment and Color Corp.

"Color Standards for Opaque Materials," by I. H. Godlove, Physicist and Chemist, Technical Laboratory, E. I. du Pont de Nemours and Co.

"Spectrophotometry and Color Evaluation," by A. E. Parker, Physicist, Electrical Testing Laboratories.

"Photoelectric Tristimulus Colorimetry," by Richard S. Hunter, Assistant Physicist, National Bureau of Standards.

The presiding officer at the symposium session and the officers representing the two sponsor bodies, were as follows:

M. Rea Paul (*Symposium Chairman*), National Lead Co.
W. M. Barr (*President, A.S.T.M.*), Chief Chemical and Metallurgical Engineer, Union Pacific Railroad Co.
Deane B. Judd (*Chairman, I.S.C.C.*), Physicist, National Bureau of Standards.
G. E. F. Lundell (*Vice-President, A.S.T.M.; Chairman,* Washington Committee on Arrangements), Chief, Chemistry Division, National Bureau of Standards.

The evening of Wednesday, March 5, was devoted to a popular session on Parade of Color sponsored by the Inter-Society Color Council with A.S.T.M. participating.

The papers in both of the above symposiums elicited discussion which contributed greatly to the information contained in the papers. The papers of the two symposiums complete with discussion have been issued by the A.S.T.M. as separate publications entitled, respectively, "Symposium on New Methods for Particle Size Determinations in the Subsieve Range," and "Symposium on Color—Its Specification and Use in Evaluating the Appearance of Materials."

The three technical sessions were very well attended, there being over 300 persons at each session.

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